INVESTIGATION IN TO TRIBO POTENTIAL OF RICE HUSK (RH) CHAR REINFORCED EPOXY COMPOSITE

A Thesis Submitted to

National Institute of Technology, Rourkela

(Deemed University)

In Partial fulfillment of the requirement for the degree of

Master of Technology

in

Mechanical Engineering

by

SHANKAR PRASAD SHUKLA

Department of Mechanical Engineering
National Institute of Technology
Rourkela -769 008 (India)
2011

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Under the guidance and supervision of

Prof. S. K. ACHARYA

Department of Mechanical Engineering
National Institute of Technology
Rourkela -769008 (India)
2011

Dedicated to

My Father

& Mother

National Institute of Technology

Rourkela

CERTIFICATE

This is to certify that the thesis entitled "**INVESTIGATION IN TO TRIBO POTENTIAL OF RICE HUSK (RH) CHAR REINFORCED EPOXY COMPOSITE** "submitted to the National Institute of Technology, Rourkela (Deemed University) by **SHANKAR PRASAD SHUKLA**, Roll No. **209ME1193** for the award of the Degree of **Master of Technology** in **Mechanical Engineering** with specialization in "**Machine Design and Analysis**" is a record of bonafide research work carried out by him under my supervision and guidance. The results presented in this thesis has not been, to the best of my knowledge, submitted to any other University or Institute for the award of any degree or diploma.

The thesis, in my opinion, has reached the standards fulfilling the requirement for the award of the degree of **Master of technology** in accordance with regulations of the Institute.

Place: Rourkela

Date:

Dr. S. K. Acharya
Associate-Professor
.Department of Mechanical Eng.
National Institute of Technology,
Rourkela - 769008

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ABSTRACT

Rice husk (RH) is an agricultural waste material abundantly available in rice-producing countries. They are the natural sheaths that from on rice grains during their growth. Removal during the refining of rice, these husks have no commercial interest. The annual rice husk production in India amounts is generally approximately 12 million tons. Rice husk is generally not recommended as cattle feed since cellulose and other sugar contents are low. Worldwide production of rice husk is about 120 million tons per year. That makes the rice husk one of the largest readily available but also one of the most under-utilized resources. Increase of environmental awareness has led to a growing interest in researching ways of an effective utilization of rice by-product, from which rice husk is particularly valuable due to its high content of amorphous silica .But it is interesting to note that rice husk contains 20% ash, 22% lignin, 38% cellulose, 18% pentosans and 2% moisture. It is felt that the value of this agricultural residue can be upgraded by bonding with resin to produce composite suitable for tribological applications.

Keeping this in view the present work has been under taken to develop a polymer matrix composite (epoxy resin) using RH char and to study its tribological behavior, the new hard porous carbon material rice husk char (RH char) has been developed by carburizing rice husk as the main raw material at three different tempreture range 850°c, 900^0c and 950°c. The composite are prepared with different volume fraction of RH Char. Experiments have been conducted under laboratory condition using pin-on-disk wear tester. All the experiments have been conducted under dry condition only with different variables. Micro structure examination (SEM) of the worn surfaces has been carried out to study the effect of RH char on wear bahaviour.

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NOMENCLATURE

Weight loss	**Δw**
Wear rate	**W**
Specific wear rate	**W_s**
Load	**N**
Sliding velocity	**V**
Volume fraction	**V_f**
Volume fraction of voids	**Vv**
Theoretical density	**ρ_t**
Actual density	**ρ_a**
Density	**ρ**
Sliding distance	**S_d**
Applied Load	**L**
Fiber volume fraction	**R_e**

Chapter 1

INTRODUCTION

CHAPTER – 1

1.1 Back ground:

The developments in composite material after meeting the challenges of aerospace sector have cascaded down for catering to domestic and industrial applications. Composites, the wonder material with light-weight; high strength-to-weight ratio and stiffness properties have come a long way in replacing the conventional materials like metals, wood etc. A unique feature of composites is that the characteristics of the finished product can be tailored to a specific engineering requirement by the careful selection of matrix and the reinforcement type.

Strictly speaking, the idea of composite materials is not a new or recent one. Nature is full of examples wherein the idea of composite materials is used. The coconut palm leaf, for example, is nothing but a cantilever using the concept of fiber reinforcement. Wood is a fibrous composite; cellulose fibers in a lignin matrix. The cellulose fibers have high tensile strength but are very flexible (i.e. low stiffness), while the lignin matrix joins the fibers and furnishes the stiffness. Bone is yet another example of a natural composite that supports the weight of various members of the body. It consists of short and soft collagen fibers embedded in a mineral matrix called apatite. Nevertheless, one can safely mark the origin of the distinct discipline of the composite materials as the beginning of the 1960s. It would not be too much off the mark to say that a concerted research and development effort in composite materials began in 1965. Since the early 1960s, there has been an increasing demand for materials that are stiffer and stronger yet lighter in fields as diverse as aerospace, energy and civil constructions.

1.2 DEFINITION OF COMPOSITE:

The most widely used meaning is the following one, which has been stated by Jartiz [1] “Composites are multifunctional material systems that provide characteristics not obtainable from any discrete material. They are cohesive structures made by physically combining two or more compatible materials, different in composition and characteristics and sometimes in form”. Accordingly one may well classify among the composite materials nearly all substances such as wood, bones, shell etc., and also some man-made materials such

as certain powder metallurgy products, electrical insulators, resin bonded magnetic materials, powder charged plastics, paper laminates etc..

The weakness of this definition resided in the fact that it allows one to classify among the composites any mixture of materials without indicating either its specificity or the laws which should give it which distinguishes it from other very banal, meaningless mixtures.

Kelly [2] very clearly stresses that the composites should not be regarded simple as a combination of two materials. In the broader significance; the combination has its own distinctive properties. In terms of strength to resistance to heat or some other desirable quality, it is better than either of the components alone or radically different from either of them.

Berghezan [3] defines as "The composites are compound materials which differ from alloys by the fact that the individual components retain their characteristics but are so incorporated into the composite as to take advantage only of their attributes and not of their short comings", in order to obtain improved materials.

VanSuchetclan [4] explains composite materials as heterogeneous materials consisting of two or more solid phases, which are in intimate contact with each other on a microscopic scale. They can be also considered as homogeneous materials on a microscopic scale in the sense that any portion of it will have the same physical property.

From the above definition, it clearly results that what has to be achieved during the design and the fabrication of a composite material, is the incorporation into its structure of a synergic effect so as to be able to obtain " a new material possessing superior properties to the individual components, either alone or mixed together".

1.3 CHARACTERISTICS OF THE COMPOSITES:

Composites consist of one or more discontinuous phases embedded in a continuous phase. The discontinuous phase is usually harder and stronger than the continuous phase and is called the 'reinforcement' or 'reinforcing material', whereas the continuous phase is termed as the ' matrix'.

Properties of composites are strongly dependent on the properties of their constituent materials, their distribution and the interaction among them. The composite properties may be the volume fraction sum of the properties of the constituents or the constituents may interact in a synergistic way resulting in improved or better properties. Apart from the nature of the constituent materials, the geometry of the reinforcement (shape, size and size distribution) influences the properties of the composite to a great extent. The concentration distribution and orientation of the reinforcement also affect the properties.

The shape of the discontinuous phase (which may by spherical, cylindrical, or rectangular cross-sanctioned prisms or platelets), the size and size distribution (which controls the texture of the material) and volume fraction determine the interfacial area, which plays an important role in determining the extent of the interaction between the reinforcement and the matrix.

Concentration, usually measured as volume or weight fraction, determines the contribution of a single constituent to the overall properties of the composites. It is not only the single most important parameter influencing the properties of the composites, but also an easily controllable manufacturing variable used to alter its properties.

1.4 CLASSIFICATION:

Composite materials can be classified in different ways [5]. Classification based on the geometry of a representative unit of reinforcement is convenient since it is the geometry of the reinforcement which is responsible for the mechanical properties and high performance of the composites. A typical classification is presented in table1.1.The two broad classes of composites are (1) Particulate composites and (2) Fibrous composites.

1.4.1 Particulate Composites:

As the name itself indicates, the reinforcement is of particle nature (platelets are also included in this class). It may be spherical, cubic, tetragonal, a platelet, or of other regular or irregular shape, but it is approximately equiaxed. In general, particles are not very effective in improving fracture resistance but they enhance the stiffness of the composite to a limited extent. Particle fillers are widely used to improve the properties of matrix materials such as to modify the thermal and electrical conductivities, improve performance at elevated

temperatures, reduce friction, increase wear and abrasion resistance, improve machinability, increase surface hardness and reduce shrinkage.

1.4.2 Fibrous composites:

A fiber is characterized by its length being much greater compared to its cross-sectional dimensions. The dimensions of the reinforcement determine its capability of contributing its properties to the composite. Fibers are very effective in improving the fracture resistance of the matrix since a reinforcement having a long dimension discourages the growth of incipient cracks normal to the reinforcement that might otherwise lead to failure, particularly with brittle matrices.

Man-made filaments or fibers of non-polymeric materials exhibit much higher strength along their length since large flaws, which may be present in the bulk material, are minimized because of the small cross-sectional dimensions of the fiber. In the case of polymeric materials, orientation of the molecular structure is responsible for high strength and stiffness.

Fibers, because of their small cross- sectional dimensions, are not directly usable in engineering applications. They are, therefore, embedded in matrix materials to form fibrous composites. The matrix serves to bind the fibers together, transfer loads to the fibers, and protect them against environmental attack and damage due to handling. In discontinuous fiber reinforced composites, the load transfer function of the matrix is more critical than in continuous fiber composites.

1.5 COMPONENTS OF A COMPOSITE MATERIAL

In its most basic form a composite material is one, which is composed of at least two elements working together to produce material properties that are different to the properties of those elements on their own. In practice, most composites consist of a bulk material (the ‘matrix’), and a reinforcement of some kind, added primarily to increase the strength and stiffness of the matrix.

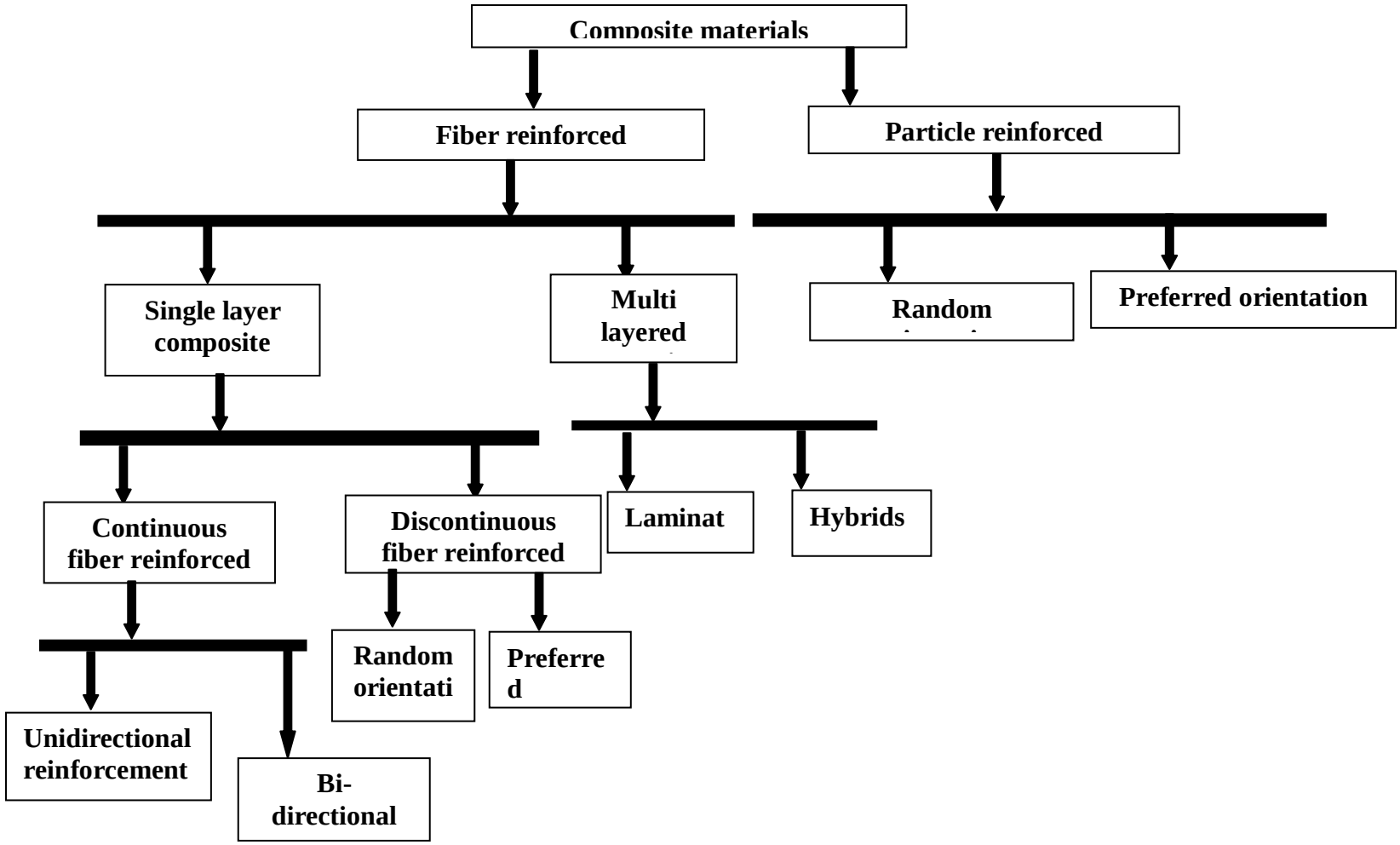


Table-1.1: Classification of composite

1.5.1 Role of matrix in a composite

Many materials when they are in a fibrous form exhibit very good strength property but to achieve these properties the fibers should be bonded by a suitable matrix. The matrix isolates the fibers from one another in order to prevent abrasion and formation of new surface flaws and acts as a bridge to hold the fibers in place. A good matrix should possess ability to deform easily under applied load, transfer the load onto the fibers and evenly distributive stress concentration.

1.5.2 Materials used as matrices in composites

In its most basic form a composite material is one, which is composed of at least two elements working together to produce material properties that are different to the properties of those elements on their own. In practice, most composites consist of a bulk material (the matrix) and a reinforcement of some kind, added primarily to increase the strength and stiffness of the matrix.

(a) BULK PHASES

(1) Metal Matrices

Metal matrix composites possess some attractive properties, when compared with organic matrices. These include (i) strength retention at higher temperatures, (ii) higher transverse strength, (iii) better electrical conductivity, (iv) superior thermal conductivity, (v) higher erosion resistance etc. However, the major disadvantage of metal matrix composites is their higher densities and consequently lower specific mechanical properties compared to polymer matrix composites. Another notable difficulty is the high-energy requirement for fabrication of such composites.

(2) Polymer Matrices

A very large number of polymeric materials, both thermosetting and thermoplastic, are used as matrix materials for the composites. Some of the major advantages and limitations of resin matrices are shown in Table 1.1.

Generally speaking, the resinous binders (polymer matrices) are selected on the basis of adhesive strength, fatigue resistance, heat resistance, chemical and moisture resistance etc. The resin must

have mechanical strength commensurate with that of the reinforcement. It must be easy to use in the fabrication process selected and also stand up to the service conditions. Apart from these properties, the resin matrix must be capable of wetting and penetrating into the bundles of fibers which provide the reinforcement, replacing the dead air spaces therein and offering those physical characteristics capable of enhancing the performance of fibers.

(3) Ceramic Matrices

Ceramic fibers, such as alumina and SiC (Silicon Carbide) are advantageous in very high temperature applications, and also where environment attack is an issue. Since ceramics have poor properties in tension and shear, most applications as reinforcement are in the particulate form (e.g. zinc and calcium phosphate). Ceramic Matrix Composites (CMCs) used in very high temperature environments, these materials use a ceramic as the matrix and reinforce it with short fibers, or whiskers such as those made from silicon carbide and boron nitride.

(b) REINFORCEMENT

The role of the reinforcement in a composite material is fundamentally one of increasing the mechanical properties of the neat resin system. All of the different fibers used in composites have different properties and so affect the properties of the composite in different ways. For most of the applications, the fibers need to be arranged into some form of sheet, known as a fabric, to make handling possible. Different ways for assembling fibers into sheets and the variety of fiber orientations possible to achieve different characteristics.

(c) INTERFACE

It has characteristics that are not depicted by any of the component in isolation. The interface is a bounding surface or zone where a discontinuity occurs, whether physical, mechanical, chemical etc. The matrix material must “wet” the fiber. Coupling agents are frequently used to improve wettability. Well “wetted” fibers increase the interface surfaces area. To obtain desirable properties in a composite, the applied load should be effectively transferred from the matrix to the fibers via the interface. This means that the interface must be large and exhibit strong adhesion between fibers and matrix. Failure at the interface (called debonding) may or may not be desirable.

1.6 TYPES OF COMPOSITE MATERIALS:

The composite materials are broadly classified into the following categories:

1.6.1 Fiber-Reinforced Composites

Reinforced Composites are popularly known being used in many industrial applications because of their inherent high specific strength and stiffness. Due to their excellent structural performance, the composites are gaining potential also in tribological applications. In this type of composite the second phase is in the form of fibers dispersed in the Matrix which could be either plastic or metal. The volume fraction (V_f) varies from a few percentage to as high as 70%. Usually the fiber reinforcement is done to obtain high strength and high modulus. Hence is necessary for the fibers to posses' higher modulus than the matrix material, so that the load is transferred to the fiber from the matrix more effectively.

1.6.1.1 Continuous or long fiber composite:

Continuous or long fiber composite consist of a matrix reinforced by a dispersed phase in form of continuous fibers. A continuous fiber is geometrically characterized as having a very high length-to- diameter ratio. They are generally stronger and stiffer than bulk material. Based on the manner in which fibers are packed within the matrix, it is again subdivided in to two categories: (a) unidirectional reinforcement and (b) bidirectional reinforcement.

1.6.1.2 Discontinuous or short fiber composite:

Short-fiber reinforced composites consist of a matrix reinforced by a dispersed phase in form of discontinuous fibers (length < 100*diameter).The low cost, ease of fabricating complex parts, and isotropic nature are enough to make the short fiber composites the material of choice for large-scale production. Consequently, the short-fiber reinforced composites have successfully established its place in lightly loaded component manufacturing. Further the discontinuous fiber reinforced composite divided into: (a) biased or preferred oriented fiber composite and (b) random oriented fiber composite.

1.6.2 Dispersion hardened Material:

In this type of material, fine particles of sizes ranging from .01μm to .14μm are dispersed in matrix. Their concentration varies from 1% to 10% by volume. These fine particles impede dislocation movement in the material and therefore result in very high strength. Also these materials possess improved high temperature strength and creep resistance.

1.6.3 Laminate Composites:

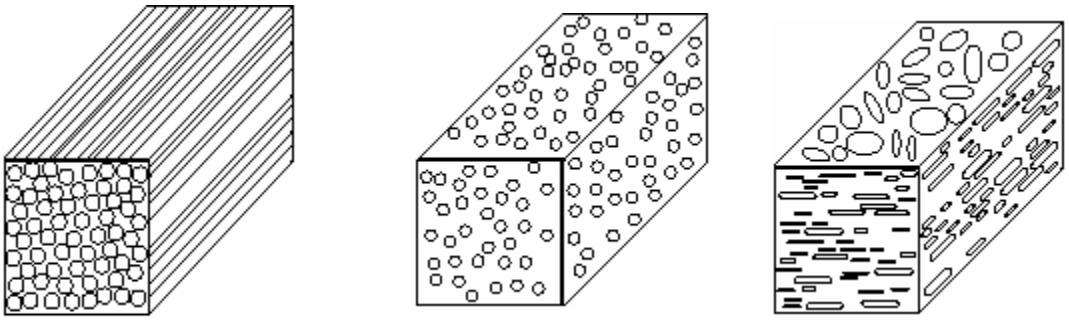
Laminate Composites are composed of layers of materials held together by matrix. Generally, these layers are arranged alternatively for the better bonding between reinforcement and the matrix. These laminates can have uni- directional or bi-directional orientation of the fiber reinforcement according to the end use of the composite.The different types of composite laminates are unidirectional, angle-ply, cross-ply and symmetric laminates.

1.6.4 Particulate Composites:

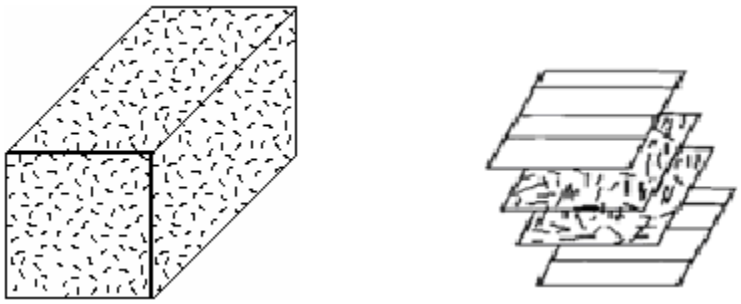
In this type of composites, 1μm to 200μm size particles are dispersed in the matrix and the volume fraction is generally between $.01V_f$ to $.85V_f$. discontinuously reinforced Aluminium (DRA) composites are a subclass of metal matrix composites. DRA is an Aluminium alloy combined with a discrete reinforcement phase (particles, whiskers or chopped fibers) in such a way as to retain the beneficial properties of each of the constituents. Their combination of properties and fabric ability makes them attractive candidates for many structural components requiring high stiffness, high strength and low weight.

1.7 APPLICATION OF COMPOSITES

The application and usage of the composites encompasses a wide variety of products and processes. These can be broadly put into following categories:



(a) Continuous fiber composite (b) Particulate composites (c) Flake composites



(d) Random fiber (short fiber) Composite (e) Laminate Composite

Fig 1.1 (a-e), Schematic of different types of Composite

1.7.1 High Performance Area:

This forms the high-tech end of the spectrum and comprises mainly of Aerospace sector with applications in air craft launch, chemicals satellites and missiles etc. The volume of consumption is low but the technological requirements are of higher order.

1.7.2 High volume/commercial area:

This consists of very large range of products in both engineering and consumer sector that are produced commercially. Main application areas are Agriculture, automotive/ transportation, pollution control and general engineering. The demand on technology level is not as high in the high performance area.

1.7.3 Speciality area:

This consists of special applications where technology level and volume may be high or low but, special consideration dictates the product development. These include Biomedical applications and other specialized areas.

1.8 Ceramic composites:

Ceramic materials are inorganic, non metallic materials and things made from them. They may be crystalline or partly crystalline. They are formed by the action of heat and subsequent cooling [6] clay was one of the earliest materials used to produce ceramics, but many different ceramic materials are now used in domestic, industrial and building products. A ceramic material may be defined as any inorganic crystalline oxide material. It is solid and inert. Ceramic materials are brittle, hard, and strong in compression, weak in shearing and tension. They withstand chemical erosion that occurs in an acidic or caustic environment. In many cases withstanding erosion from the acid and bases applied to it.

Ceramics generally can withstand very high temperatures such as temperatures that range from 900 °C to 1,600 °C. Exceptions include inorganic materials that do not have oxygen such as silicon carbide. Glass by definition is not a ceramic because it is an amorphous solid (non-crystalline). However, glass involves several steps of the ceramic process and its mechanical properties behave similarly to ceramic materials.

Traditional ceramic raw materials include clay minerals such as kaolinite, more recent materials include aluminium oxide, more commonly known as alumina. The modern ceramic materials, which are classified as advanced ceramics, include silicon carbide and tungsten carbide. Both are valued for their abrasion resistance, and hence find use in applications such as the wear plates of crushing equipment in mining operations. Advanced ceramics are also used in the medicine, electrical and electronics industries.

Ceramic materials are usually ionic or covalent bonded materials, and can be crystalline or amorphous. A material held together by either type of bond will tend to fracture before any plastic deformation takes place, which results in poor toughness in these materials. Additionally,

because these materials tend to be porous, the pores and other microscopic imperfections act as stress concentrations, decreasing the toughness further, and reducing the tensile strength. These combine to give catastrophic failures, as opposed to the normally much more gentle failure modes of metals.

These materials do show plastic deformation. However, due to the rigid structure of the crystalline materials, there are very few available slip systems for dislocations to move, and so they deform very slowly. With the non-crystalline (glassy) materials, viscous flow is the dominant source of plastic deformation, and is also very slow. It is therefore neglected in many applications of ceramic materials.

Silicon carbide is extremely hard and has a high thermal conductivity, high thermal-shock resistance, high hot strength, high melting point, a low coefficient of thermal expansion, good oxidization resistance and good corrosion resistance to acid and base. By all the above properties SiC is used as fiber in preparation of composites in the past two decades. The strength and hardness of the ceramics greatly enhanced by the addition of SiC particles into the matrixes,

1.8.1 Rice husk-Initiative in product development:

Rice husk (RH) is an agricultural waste material abundantly available in rice-producing countries. They are the natural sheaths that from on rice grains during their growth. Removed during the refining of rice, these husks have no commercial interest [7]. Globally, approximately 600 million tons of rice paddy is produced each year. On average 20% of the rice paddy is husk, giving an annual total production of 120 million tones. A large quantity of husk, which is known to have a fibrous material with high silica content, is available as waste from rice milling industries.

The treatment of rice husk as a ‘resource’ for energy production is a departure from the perception that husks present disposal problems. Rice husk is unusually high in ash compared to other biomass fuels – close to 20%. The ash is 92 to 95% silica, highly porous and lightweight, with a very high external surface area. Its absorbent and insulating properties are useful to many research studies. The chemical composition of the rice husk ash varies from sample to sample which may be due to the different geographical conditions, type of paddy, climatic conditions and type of fertilizer used [8].

However it is interesting to note that rice husk generally contains 20% ash, 22% lignin, 38% cellulose, 18% pentosans and 2% moisture. RHA is a general term describing all types of ash produced from burning rice husks. In practice, the type of ash varies considerably according to the burning technique.

Silicon enters the rice plant through its root in a soluble form, probably as a silicate or monosilicic acids, and then moves to the outer surface of the plant, where it is become concentrated by evaporation and polymerization to form a cellulose silica membrane. There is quite general agreement that the silica is predominantly in inorganic linkages, but some of the silica is also bonded covalently to the organic compounds. This portion of the silica cannot be dissolved in alkali and can withstand very high temperatures [9].

In general, rice husk ash (RSA) might well be considered slightly impure silica. The content of silica and all impurities in RHA vary depending on the variety, climate and geographic location [10].

RHs contain mainly 15-20 wt% silica and a number of organic constituents that will yield carbon when thermally decomposed. Therefore RHA contains two necessary raw materials for the preparation of silicon carbide: SiO_2 and C. with the very high surface area and intimate content available for the carbon and silica in RHs, it is possible to form SiC at relatively low temperature [11] (much lower than indicated by thermodynamic and kinetic calculations) [12], in addition , the silica in the RHs can maintain the initial structure of the RHs. Both the low density and the space in the raw materials facilitate the production of silicon carbide [13] therefore RHs are the most economical and promising raw material for the producing silicon carbide.

Silica not only is an important starting material for semiconductors but also plays an important role in the plastics, rubber, and photoelectric material industries. [14] RHs can be directly combusted to produce silica without pretreatment [15-19]. The silica in the ash undergoes structural transformations depending on the conditions (time, temperature etc.) of combustion. At 550°C – 800°C amorphous ash is formed and at temperatures greater than this, crystalline ash is formed. These types of silica have different properties and it is important to produce ash of the correct specification for the particular end use.

- This study is focused on the carburization of RICE HUSK.
- Carburization is defined as the extensive thermal degradation of the carbonaceous component in the absence of air, or in the presence of inert gas. During this process, the volatiles are removed in the form of oxides of carbon and hydrogen to enrich the carbon content in the solid char [20].

1.8.2 Applications of rice husk:

1.8.2.1 Cement and concrete:

Substantial research has been carried out on the use of amorphous silica in the manufacture of concrete. There are two areas for which RHA is used, in the manufacture of low cost building blocks and in the production of high quality cement.

1.8.2.2 Refractory bricks:

Due to its insulating properties, RHA has been used in the manufacture of refractory bricks [20].Refractory bricks are used in furnaces which are exposed to extreme temperatures, such as in blast furnaces used for producing molten iron and in the production of cement clinker.

1.8.2.3 Lightweight construction materials:

There is anecdotal evidence of RHA being used in the manufacture of lightweight insulating boards in developing countries [20]. Research at the University of Arkansas has also focused the manufacture of insulation from RHA (Plate 8).

1.8.2.4 Silicon chips:

The first step in semi-conductor manufacture is the production of a wafer, a thin round slice of semi-conductor material, which is usually silicon. Purified polycrystalline silicon (traditionally created from sand) is heated to a molten liquid and a small piece of silicon (seed) placed in the molten liquid. As the seed is pulled from the melt the liquid cools to form a single

crystal ingot. This is then ground and sliced to form wafers which are the starting material for manufacturing integrated circuits [21, 22].

1.8.2.5 Brake Pad reinforcement material:

There is very little information on the use of RHA in brake pad, other than that it must be pure and high quality [23].

1.8.2.6 Oil absorbent:

Husks burnt slowly over a period of six months have been found to be effective as oil absorbent and are marketed in California under the trade name 'Greasweep' (Plate 9). This is a relatively small operation, but there is potential to increase this market. It is thought it is amorphous ash that is being used [24].

There are other uses for RH which are still in the research stages [21]:

- In the manufacture of roof tiles
- As a free running agent for fire extinguishing powder
- An abrasive filler for tooth paste
- A component of fire proof material and insulation
- As a beer clarifier
- Extender filler for paint
- Production of sodium silicate films [25, 26]

It is known that RH char contains amorphous silica in addition to amorphous carbon as the main constituents. Compares to the conventional sources of silica, rice husk ash as a char raw material possess advantages like fine particles size and higher reactivity due to its amorphous nature. Many authors have concluded that rice husk is an excellent source of high grade amorphous silica [27-31]. Accordingly silica is used in different industries and products like rubber industry as a reinforcing agent, in tooth pastes as a cleaning agent, as an anti-caking agent in salts, in cosmetics etc.

But it is interesting to note that Silica obtained from rice husk ash is highly reactive, depending upon the degree of firing and is used for making insulating materials, refractory bricks, Portland cement, masonry cement and pottery ware. It is used as filler materials in paints and in fertilizers etc. compared with silicon-carbide-reinforced metal matrix composites and ceramic matrix composites, the research on silicon-carbide-reinforced resin matrix composites is limited. Considering the high price of the carbon fibers that are used in resin composites, cheap silicon carbide whiskers from RHs could be a suitable substitute for them. Another advantage of using silicon carbide whiskers as reinforcement lies in the low requirement on the purity of the silicon carbide whiskers from RHs are carbon, silica, and silicon carbide particles, all of which have little negative effect on resin composites' mechanical properties. [32].

Recently, Wang et al. [33] reported the synthesis of penta coordinate silicon complexes from RHA. They are easily hydrolyzed to silica. Such complexes can also be reacted with other reactants to prepare thermally stable products. They can also be reacted in situ with SiO_2 or Al_2O_3 to prepare ceramic matrix composites.

When silicon carbide is used as an abrasive, the requirement on purity is not high. The purity of silicon carbide from RHs is high enough to match this requirement. The silicon carbide can be either directly used as abrasive material or hot-pressed with composites. [34]

Visualizing the increased rate of utilization of rice husk, in the present work the new hard porous carbon material called the RH (rice husk) Char an attempt has been made to develop. Out of the available manufacturing procedures we have adopted the usual stir casting technique to prepare the composite. Different volume fraction of rice husk char has been mixed with the matrix material and specimens were prepared for wear studies. The wear studies have been carried out using a pin-on-disc wear-testing machine under simulated laboratory conditions. All the experiments have been conducted in dry conditions only, with different variables.

In the second chapter detailed discussion on selection of epoxy as the matrix material, reinforcement material and work related to present investigations available in literatures are presented.

Chapter 2

LITERATURE SURVEY

CHAPTER – 2

2. 1INTRODUCTION

The literature survey is carried out as a part of the thesis work to have an overview of the production processes, properties and weathering behavior of polymer matrix composites. Composite structures have shown universally a savings of at least 20% over metal counterparts and a lower operational and maintenance cost [35]. As the data on the service life of composite structures is becoming available, it can be safely said that they are durable, maintain dimensional integrity, resist fatigue loading and are easily maintainable and repairable. Composites will continue to find new applications, but the large scale growth in the marketplace for these materials will require less costly processing methods and the prospect of recycling [36] will have to be solved [37].

Composites materials have emerged as a major class of structural elements and are either used or being considered as substitutions for metals/traditional material in aerospace, automotive and other industries. The outstanding features of fiber reinforced polymer composites (FRPs) are their high specific stiffness, high specific strength and controlled anisotropy, which make them very attractive structural materials. Other advantages of composites are light weight, good corrosion resistance, impact resistance, fatigue strength and flexibility in design capabilities. A unique feature of composites is that the characteristics of the finished product can be tailored to a specific engineering requirement by a careful selection of matrix and reinforcement type. FRP composite materials consist of two or more chemically distinct constituents have a distinct interface separating them. It has a unique combination of properties that are noticeably different from the constituent properties. Generally, a discontinuous phase (reinforcement) is embedded into a continuous phase (matrix). Polymer based composite materials (PMC) or FRP constitutes a major category of composites materials with a wide range of applications. They offer very attractive properties, which can be tailored to the specific requirements by careful selection the fiber, matrix, fiber configuration (short, long, strength, woven, braided, laminated, etc.) and fiber surface treatment. PMCs exhibit desirable physical and chemical properties that include lightweight coupled with high stiffness and strength along the direction of the reinforcing fiber,

dimensional stability, temperature and chemical resistance and relatively easy processing. The role of matrix in a fiber-reinforced composite is to

(a) Transfer stresses between the fibers
(b) Provide a barrier against an adverse environment
(c) Protect the surface of fibers from mechanical abrasion.

2.2MATERIAL SELECTION:

2.2.1 Matrix Material:

Because it is much more than dispersing glue in PMC, the matrix alloy should be chosen only after giving careful consideration to its chemical compatibility with the reinforcement, to its ability to wet the reinforcement, and to its own characteristics properties and processing behavior [36, 38].

2.2.2 Why Polymer Matrix Selection?

Polymers are structurally much more complex than metals or ceramic. They are cheap and can be easily processed. On the other hand, polymers have lower strength and modulus and lower temperature use limits. Prolonged exposure to ultraviolet light and some solvents can cause the degradation of polymer properties. Because of predominantly covalent bonding, polymers are generally poor conductors of heat and electricity. Polymers, however, are generally more resistant to chemicals than are metals. Structurally, polymers are giant chainlike molecules (hence the name macromolecules) with covalently bonded carbon atoms forming the backbone of the chain. The process of forming large molecules from small ones is called polymerization; that is, polymerization is the process of joining many monomers, the basic building blocks, together to form polymer. Polymers used to manufacture advanced PMCs are of two basic types thermoset and thermoplastics resins.

a) Thermoset resins:

Thermoset resins dominate the advanced composites industry today, while thermoplastics have only a minor role. It requires addition of a curing agent or hardener and impregnation onto a reinforcing material, followed by a curing step to produce a cured or finished part. Some of the more common thermoset resins are described briefly here.

Epoxy resins are relatively low molecular weight monomers with low shrinkage during cure. They can be partially cured and stored in that state. The cured epoxy resins have high chemical and corrosion resistance, good mechanical thermal properties. However, they are more expensive compared to polyester resin. The second of the essential ingredients of an advanced composite system is the curing agent or hardner. These compounds are very important because they control the reaction rate and determine the performance characteristics of the finished part. Since these compounds act as catalysts for the reaction, they must contain active sites on their molecules. Some of the most commonly used curing agent in the advanced composite industry is the aromatic amines like 4.4 methylene-di-aniline (MDA) and 4.4-sulfonylianiline (DDS)

Unsaturated polyester resins are most widely used due to their good mechanical properties, corrosion resistance, low weight and low cost. These consist of linear polymer chains dissolved in styrene monomer. These polymer chains have reactive sites resulting from the incorporation of the anhydrous forms of unsaturated di carboxylic acids (e.g. maleic anhydride). The reactive unsaturation sites on the polymer chains react and crosslinking with the styrene monomer via a free radical reaction. This reaction is usually initiated by the addition of a peroxide catalyst, such a methyl ethyl ketone peroxide (MEKP). The presence of an accelerator in the resin, such as cobalt octate, speed the reaction at a given temperature. The addition of heat significantly speeds up this cross-linking reaction.

Polyurethanes are another group of resin used in advanced composite process. These compounds are formed by reaction the polyol component with an isocynate compound, typically toluene diisocynate (TDI). Methylene diisocynate (MDI) and hexamethylenediisocynate (HDI) are also widely used. These are used to manufacture fiber reinforced structural foams. Phenolic

and amino resins are used extensively in aircraft interiors because of the exceptional low smoke and heat release properties in the event of a fire.

b) Thermoplastic resins:

Thermoplastics resins require only heat and pressure to form the finished part. Unlike the thermoset resins, the thermoplastics resins can usually be reheated and reformed into another shape, if desired. Common examples of thermoplastics resins are polyethylene, polystyrene, nylon, polycarbonate, polysulfone, polyphenylenesulfine etc. Polyamides, Polyimide, PEEK are relatively newcomers to the composite industry and are used for high temperature applications. These resins have better thermal stability and flame resistance than the epoxy resins. Polyamide based composites have excellent retention of strength in how-wet environment but they are brittle and have a very low elongation at break.

2.2.3 Reinforcement:

Reinforcement increases the strength, stiffness and the temperature resistance capacity and lowers the density of PMC. In order to achieve these properties the selection depends on the type of reinforcement, its method of production and chemical compatibility with the matrix and the following aspects must be considered while selecting the reinforcement material.

- Size – diameter and aspect ratio
- Shape – Chopped fiber, whisker, spherical or irregular particulate, flake, etc:
- Surface morphology – smooth or corrugated and rough:
- Poly – or single crystal
- Structural defects – voids, occluded material, second phase
- Surface chemistry
- Impurities
- Inherent properties – strength, modulus and density.

2.2.4 Reinforcement Materials:

Fibers as reinforcing material offer two advantages. Firstly, the bulk material is always stronger when produced as small diameter fibers due to the natural exclusion of large scale defects. Secondly, the fiber configuration allows the tailoring of properties in specific directions. Fibers are added to the resin system to provide strength to the finished part. The selection of reinforcing material is based on the properties desired in the finished product. Fibers used in advanced composite manufacture come in various forms, such as yarns, roving, chopped strands, woven fabric and mats. Each of these has its own special application. In processes such as filament winding or pultrusion, yarns and roving are used. When performs are used in parts manufacture, woven fabric or mats are required.

2.3 FABRICATION METHODS OF PMCs:

There are two general divisions of composites manufacturing processes: open molding and closed molding.With open molding, the gel coat and laminate are exposed to the atmosphere during the fabrication process. In closed molding, the composite is processed in a two-part mold set, or within a vacuum bag. There are a variety of processing methods within the open and closed molding categories:

a) Open Molding Method: Hand Lay-Up, Spray-Up, Filament Winding

b) Closed Molding Method: Compression molding, Pultrusion, Vacuum Bag Molding, Vacuum Infusion Processing, Resin Transfer Molding (RTM)

2.3.1 Open Molding:

Open molding process is saturating fiber reinforcement with resin, using manual rollout techniques to consolidate the laminate and removing the entrapped air. A major factor in this operation is the transfer of resin from a drum or storage tanks to the mold. The means used to transport the resin, in many cases, characterizes the specific process method.

a) Hand Lay –Up:

Hand lay-up is an open molding method suitable for making a wide variety of composites products including: boats, tanks bathware, housings, truck/auto components, architectural products and many other products ranging from very small to very large. Production volume per mold is low; however, it is feasible to produce substantial production quantities using multiple molds. Simple, single-cavity molds of fiberglass composites construction are generally used.

Moulds can range from very small to very large and are low cost in the spectrum of soft composites moulds.

Gel coat is first applied to the mold using a spray gun for a high-quality surface. When the gel coat has cured sufficiently, roll stock fiberglass reinforcement is manually placed on the mold. The lamination resin is applied by pouring, brushing, spraying, or using a paint roller. FRP rollers, paint rollers, or squeegees are used to consolidate the laminate, thoroughly wetting the reinforcement, and removing entrapped air. Subsequent layers of fiberglass reinforcement are added to build laminate thickness (Fig 2.1).

Simplest method offering low-cost tooling, simple processing and wide range of part sizes are the major advantages of this process. Design changes are readily made. There is a minimum investment in equipment. With skilled operators, good production rates consistent quality is obtainable.

b) Spray Lay-Up:

Spray-up or chopping is similar to hand lay-up in its suitability for making boats, tanks, transportation components and tub/shower units in a large variety of shapes and sizes. A chopped laminate has good conformability and is sometimes faster than hand lay-up in molding complex shapes. In the spray-up process the operator controls thickness and consistency, therefore the process is more operator dependent than hand lay-up. Although production volume per mold is low, it is feasible to produce substantial production quantities using multiple molds. As with hand lay-up, gel coat is first applied to the mold prior to spray-up of the substrate laminate.

Continuous strand glass roving and catalyzed resin are fed through a chopper gun, which deposits the resin-saturated “chop” on the mold as shown in fig 2.2. The laminate is then rolled to thoroughly saturate the glass strands and compact the chop. Additional layers of chop laminate are added as required for thickness.

c) Filament Winding:

Filament winding is an automated open molding process that uses a rotating mandrel as the mold. The male mold configuration produces a finished inner surface and a laminated rough surface on the outside diameter of the product. Filament winding results in a high degree of fiber loading, which provides high tensile strengths in the manufacture of hollow, generally cylindrical products such as chemical and fuel storage tanks, pipes, stacks, pressure vessels, and rocket motor cases. Mandrels of suitable size and shape, made of steel or aluminium form the inner surface of the hollow part. Some mandrels are collapsible to facilitate part removal.

Figure 2.3 shows the schematic picture of a typical filament winding process. Continuous strand roving is fed through a resin bath and would onto a rotating mandrel. The roving feed runs on a trolley that traverses the length of the mandrel. The filament is laid down in a predetermined geometric pattern to provide maximum strength in the directions required. When sufficient layers have been applied, the laminate is cured on the mandrel. The molded part is then stripped from the mandrel. Equipment is available for filament winding on a continuous basis with two axes winding for pressure cylinders. This process makes high strength-to-weight ratio laminates and provides a high degree of control over uniformity and fiber orientation. The filament winding process can be used to make structures, which are highly engineered and meet strict tolerances. Because filament winding is automated, the labor factor for filament winding is lower than other open molding processes.

2.3.2 Closed Molding Method:

a) Compression Molding:

Compression molding is a high-volume, high-pressure method suitable for molding complex, fiberglass-reinforced plastic parts on a rapid cycle time. There are several types of

compression molding including: sheet molding compound (SMC) which are, bulk molding compound (BMC), thick molding compound (TMC), and wet lay-up compression molding.

Compression molding tooling consists of heated metal molds mounted in large presses. Tooling is usually machined steel or cast alloy molds that can be in either single or multiple-cavity configurations. Steel molds are hardened and sometimes chrome plated for enhanced durability. The molds are heated using steam, hot oil, or electricity. Side cores, provisions for inserts, and other refinements are often employed. Mold materials include cast of forged steel, cast iron, and cast aluminum.

The mold set is mounted in a hydraulic or mechanical molding press. The molds are heated to 2500 to 4000 F. A weight charge of molding compound is placed in the open mold as shown in fig 2.4. The two halves of the mold are closed and pressure is applied. Depending on thickness, size, and shape of the part, curing cycles range from less than a minute to about five minutes. The mold is opened and the finished part is removed. Typical parts include: automobile components, appliance housings and structural components, furniture, electrical components, and business machine housings and parts.

Compression molding produces fast molding cycles and high part uniformity. The process can be automated. Good part design flexibility and features such as inserts, ribs, bosses, and attachments can be molded in .Good surface finishes are obtainable, contributing to lower part finishing cost. Subsequent trimming and machining operations are minimized in compression molding.

b) Pultrusion:

Pultrusion is a continuous process for the manufacture of products having a constant cross section, such as rod stock, structural shapes, beams channels, pipe, tubing, fishing rods, and golf club shafts. Pultrusion produces profiles with extremely high fiber loading, thus pultruded products have high structural properties. Hardened steel dies are machined and include a perform area to do the initial shaping of the resin- saturated roving. The dies include heating which can be electric or hot oil. The latest pultrusion technology uses direct injection dies, in which the resin is

introduced inside the die, rather than through an external resin bath, which may be called as partial RTM.

Continuous strand fiberglass roving, mat, cloth, or surfacing veil is impregnated in a resin bath, then pulled (pul-trusion) through a steel die, by a powerful tractor mechanism (Refer fig 2.5). The steel die consolidates the saturated reinforcement, sets the shape of the stock, and controls the fiber/resin ratio. The die is heated to rapidly cure the resin. Many creels (balls) of roving are positioned on a rack, and a complex series of tensioning devices and roving guides direct the roving into the die.

The process is a continuous operation that can be readily automated. It is adaptable to both simple and complex cross-sectional shapes. Very high strengths are possible due to the fiber loading and labor costs are low.

c) Vacuum Bag Molding:

The mechanical properties of open-mold laminates can be improved with vacuum bagging. By reducing the pressure inside the vacuum bag, external atmospheric pressure exerts force on the bag. The pressure on the laminate removes entrapped air, excess resin, and compacts the laminate. Vacuum bagging can be used with wet-lay laminates and prepreg advanced composites. In wet lay-up bagging the reinforcement is saturated using hand lay-up, then the vacuum bag is mounted on the mold and used to compact the laminate and remove air voids. In the case of pre-impreg advanced composites molding, the prepreg material is laid-up on the mold, the vacuum bag is mounted and the mold is heated or the mold is placed in an autoclave that applies both heat and external pressure, adding to the force of atmospheric pressure. The prepreg-vacuum bag-autoclave method is most often used to create advanced composites used in aircraft and military products. Molds are similar to those used for conventional open-mold processes.

In the simplest form of vacuum bagging, a flexible film (PVA, nylon, mylar, or polyethylene) is placed over the wet lay-up, the edges sealed, and a vacuum drawn. A more advanced form of vacuum bagging places a release film over the laminate, followed by a bleeder

ply of fiberglass cloth, non-woven nylon, polyester cloth, or other material that absorbs excess resin from the laminate. Fig 2.6 shows the schematic picture of vacuum bag molding process. A breather ply of a non-woven fabric is placed over the bleeder ply, and the vacuum bag is mounted over the entire assembly. Pulling a vacuum from within the bag uses atmospheric pressure to eliminate voids and force excess resin from the laminate. The addition of pressure further results in high fiber concentration and provides better adhesion between layers of sandwich construction. When laying non-contoured sheets of PVC foam or balsa into a female mold, vacuum bagging is the technique of choice to ensure proper secondary bonding of the core to the outer laminate.

Vacuum bag processing can produce laminates with a uniform degree of consolidation, while at the same time removing entrapped air, thus reducing the finished void content. Structures fabricated with traditional hand lay-up techniques can become resin rich and vacuum bagging can eliminate the problem. Additionally, complete fiber wet-out can be accomplished if the process is done correctly. Improved core-bonding is also possible with vacuum bag processing.

d) Vacuum Infusion Processing:

Vacuum infusion is a variation of vacuum bagging where the resin is introduced into the mold after the vacuum has pulled the bag down and compacted the laminate. The method is defined as having lower than atmospheric pressure in the mold cavity. The reinforcement and core material are laid-up dry in the mold. This is done by hand and provides the opportunity to precisely position the reinforcement. When the resin is pulled into the mold the laminate is already compacted; therefore, there is no room for excess resin. Very high resin to glass ratio are possible with vacuum infusion and the mechanical properties of the laminate are superior. Vacuum infusion is suitable to mold very large structures and is considered a low volume molding process. Molds are similar to those used for conventional open-mold processes.

The mold may be gel coated in the tradition fashion. After the gel coat cures, the dry reinforcement is positioned in the mold. This includes all the plies of the laminate and core material if required. A perforated release film is placed over the dry reinforcement. Next a flow

media consisting of a course mesh or a “crinkle” ply is positioned, and perforated tubing is positioned as a manifold to distribute resin across the laminate. The vacuum bag is then positioned and sealed at the mold perimeter. A tube is connected between the vacuum, bag and the resin container. A vacuum is plied to consolidate the laminate and the resin is pulled into the mold (fig 2.7).

Vacuum infusion can produce laminates with a uniform degree of consolidation, producing high strength, lightweight structures. This process uses the same low cost tooling as open molding and requires minimal equipment. Very large structures can be fabricated using this method. Vacuum infusion offers a substantial emissions reduction compared to either open molding or wet lay-up vacuum bagging.

e) Resin Transfer Molding:

Resin transfer molding is an intermediate volume molding process for producing composites. The RTM process is to inject resin under pressure into a mold cavity. Vacuum assist can be used to enhance resin flow in the mold cavity. RTM can use a wide variety of tooling, ranging from low cost composite molds to temperature controlled metal tooling. RTM can utilize either “hard” or “soft” tooling, depending upon the expected duration of the run. Soft tooling would be either polyester or epoxy molds, while hard tooling may consist of cast machined aluminum, electroformed nickel shell, or machined steel molds. RTM can take advantage of the broadest range of tooling.

Figure 2.8 shows the picture of resin transfer molding process of polyester resin with peroxide catalyst. The mold set is gel coated conventionally, if required. The reinforcement (and core material) is positioned in the mold and the mold is closed and clamped. The resin is injected under pressure, using mix/meter injection equipment, and the part is cured in the mold. The reinforcement can be either performs or pattern cut roll stock material. Performs are reinforcement that is pre-formed in a separate process and can be quickly positioned in the mold. RTM can be done at room temperature; however, heated molds are required to achieve fast cycle times and product consistency.

This closed molding process produces parts with two finished surfaces. By laying up reinforcement material dry inside the mold, any combination of materials and orientation can be used, including 3-D reinforcements. Part thickness is determined by the tool cavity.

Fiber reinforced composites are popularly being used in many industrial applications because of their high specific strength and stiffness. Due to their excellent structural performance, these composites are gaining potential also in tribological applications [39]. In this type of composites the second phase is in the form of fibers dispersed in the matrix which could be either plastic or metal. Usually the fiber reinforcement is done to obtain high strength and high modulus. Hence it is necessary for the fibers to possess higher modulus than the matrix material, so the load is transferred to the fiber from the matrix more effectively. Natural fibers to the maximum extent fulfill these criteria and therefore have drawn worldwide attention as a potential reinforcement material for the composites.

Natural fibers currently used as reinforcements in composite materials include rice husk jute, sisal, pineapple, abaca and coir [40-49]. The abundance and low cost of natural fibers combined with their low density and reduced wear on processing machinery makes these fibers suitable for use in composite materials. Synthetic fibers such as carbon or glass fibers have constant diameters; smooth surfaces and considerable rigidity. On the other hand, natural fibers can be flexible, have variable diameters along the length of each fiber and have rough surfaces. Natural fibers are also sensitive to temperature and moisture and usually have irregular cross section.

The main chemical constituents of rice husk are 20% ash, 22% lignin, 38% cellulose, 18% pentosans, 2% moisture. A typical composition of the ash is 955 SiO_2, 1% K_2O, 1% CaO, P_2O_5 and Na_2O_3, Fe_2O_3 and MgO.

The potential use of rice husk ash in cement for low cost housing has been carried out all over the world. Studies shows that replacement of 50% of Portland cement with RHA is effective and the resulting concrete cost could be 25% less [50]. Silica obtained from RHA is highly reactive, depending upon the degree of firing and is used for making insulting materials, refractory bricks [51, 52]

Andersson. C.-H.*et al.* [53]Successfully prepared relatively in expensive high grade SiC whiskers from rise husks, Way *et al.* [54,55] while worked with this SiC whiskers found that with many other ceramic materials available, this silicon carbide has a relatively high thermal conductivity and low coefficient of thermal expansion; giving it a relatively variable thermal shock resistance. Generally, the reinforcing and roughening effects of SiC whiskers are better than those of SiC particles. The proper aspect ratio is 30 – 40, as whiskers that are too long are hard to distribute. As reported by An, Z.*et al.* [56], a high content of whiskers is preferred in the production of SiC from rice husks. They also reports that thick whiskers are suitable for ceramic matrix composites; whereas thin whiskers are suitable for metal matrix composites.

Gu. Y. W. *et al.* [57] reports that upon being reinforced by silicon carbide whiskers a ceramic's fracture toughness can be greatly enhanced and its flexural strength and thermal conductivity can also be greatly improved. However Liu. Y. X. Zhao. C. X.[58] have the opinion that whenever the whiskers are being introduced into the ceramics, attention must be paid to avoid agglomeration which might result in structural defects, Wang *et al.* [59] have successfully prepared silicon carbide whiskers from rice husks and have reported that it has good reinforcing properties in ceramic matrix composites.

Some studies are also available [59-61] on aluminium reinforced by silicon carbide from rice husk. They reports that the reinforced aluminium not only has a good combination of room temperature specific strength and modulus and excellent thermal stability, but it also can be processed by normal metal working technique. Such materials are increasingly considered for aerospace applications where high stiffness and strength to weight ratios are additional advantages

Prasad B. K, *et al.* [3428] while trying to find out the factors controlling the abrasive wear of Zinc-based alloy silicon carbide particle composite has reports that when silicon carbide is used as an abrasive, the requirement on purity is not high. The purity of silicon carbide from RHs is high enough to match this requirement. They also reports that the silicon carbide can be either directly used as abrasive material or not pressed with composites.

The applications of RHA as a filler in plastics is relatively limited mainly to polypropylene (PP). As reported [62-64] with an increase in the RHA loading, a PP composites

flexural modulus and density increases, where as its tensile strength, breaking elongation and impact strength decreases, yet RHA still can replace some commercial fillers.

Navinchand *et al.* [65] reported the studies on polyester filled with RHA. Their reports say that both the tensile and impact strength of the resulting composites decreased with increasing filler loading. It is also reported that [66, 67] in addition to being used in rubbers or plastics, RHA can also be used as a filler in rubber/plastic blends.

Rozman H. D. *et al.* [68] studied the effect of chemical modification of rice husk and reports that with chemical modification the reinforcing effect can be increased to a acceptable limit.

Recently Silvia Luciana Favaro *et al.* [69] studied the chemical, morphological and mechanical analysis of rice husk /post-consumer polyethylene composites. PE and rice husk were chemically modified to improve their compatibility in composite preparation. They found improved fiber surface adhesion with matrix and improved mechanical performance compared to pure polymer matrix, on the other hand no benefit is observed in the tensile strength over the pure PE.

After reviewing the existing literature available on natural fiber composites, particularly rice husk composites efforts are put to understand the basic needs of the growing composite industry. The conclusions drawn from this is that, the success of combining rice husk ash with polymer matrices results in the improvement of mechanical properties of the composites compared with the matrix materials. These fillers are cheap and nontoxic, can be obtained from renewable sources, and are easily recyclable. Moreover, despite their low strength, they can lead to composites with high specific strengths because of their low density.

Thus the priority of this work is to prepare polymer Matrix Composites (PMCs) using rice husk char (waste from rice mill industry) as reinforcement material. A new hard porous carbon material called rice husk char has been developed and the tribological property of the RH Char reinforced epoxy composite has been studied using a pin-on-disc wear tester.

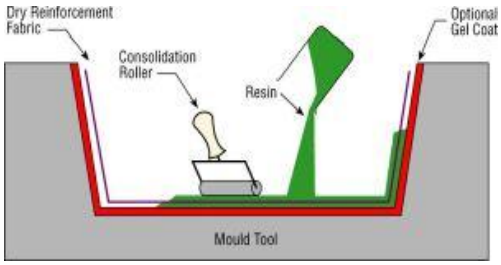


Figure 2.1 Hand Lay-Up Techniques

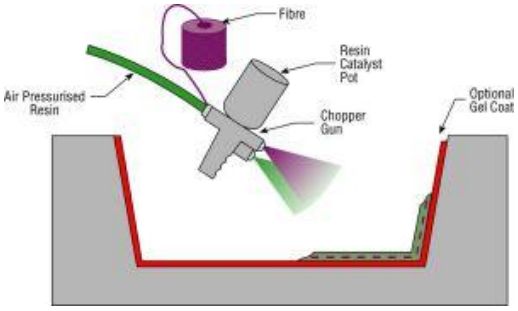


Figure 2.2 Spray up Technique

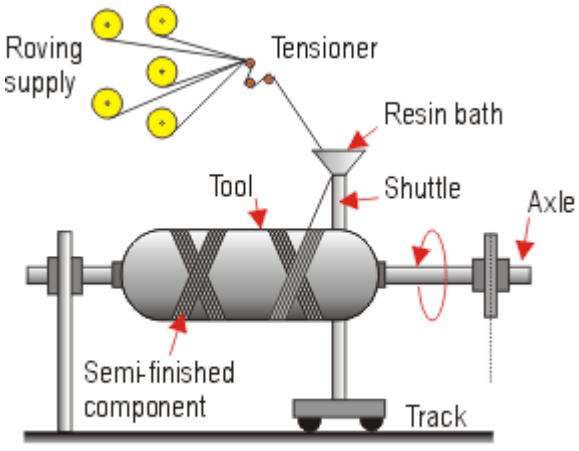


Figure 2.3 Filament Winding Process

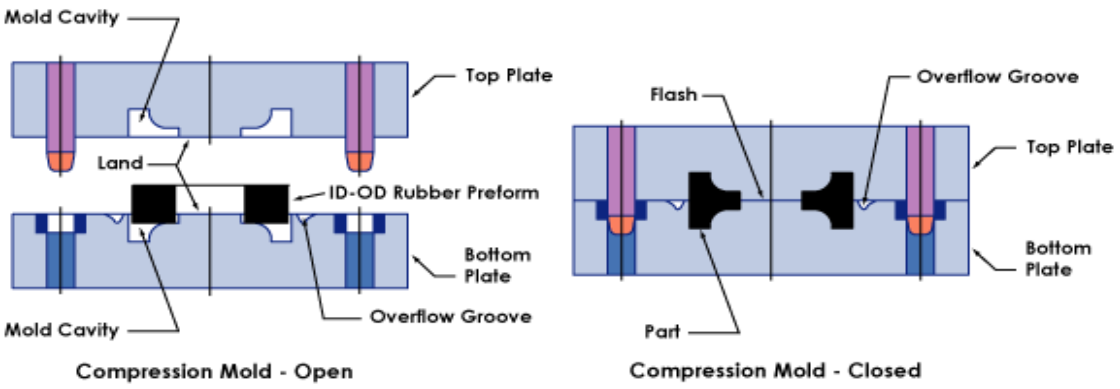


Figure 2.4 Compression Molding Technique

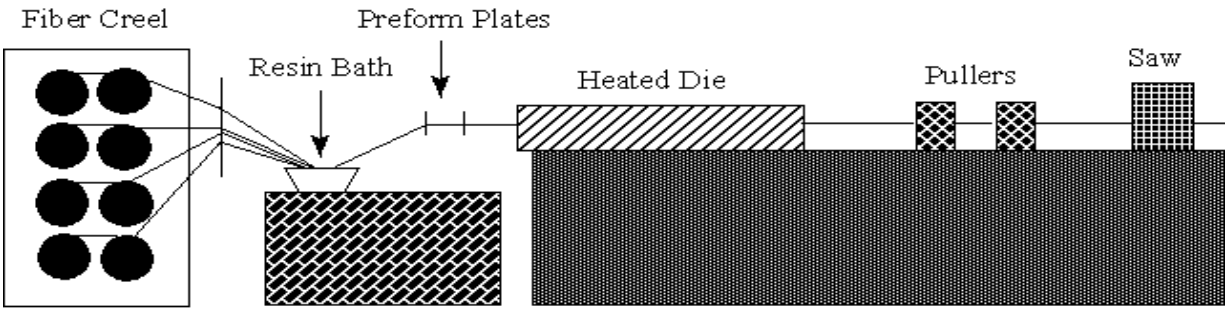


Figure 2.5 Pultrusion Process

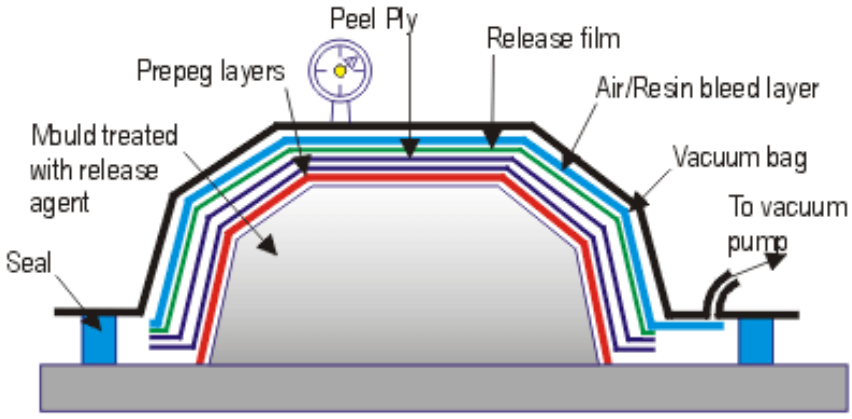


Figure 2.6 Vacuum Bag Molding

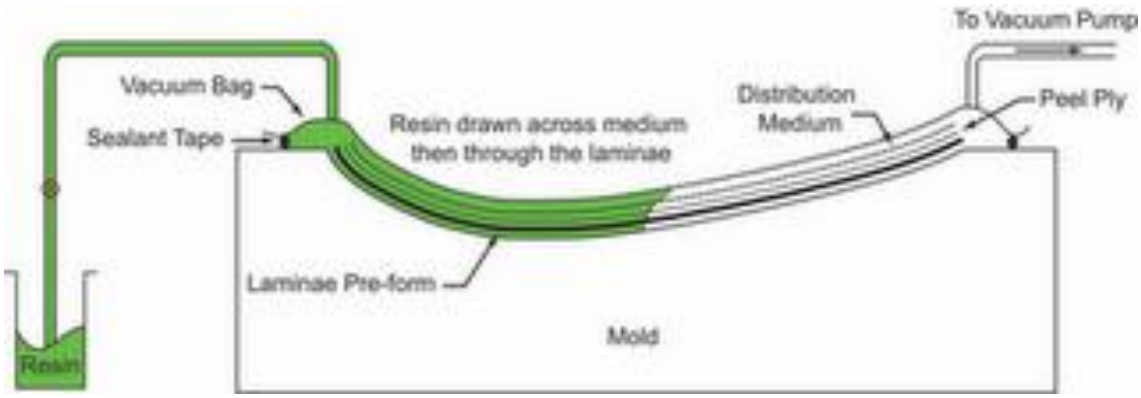


Figure 2.7 Vacuum Infusion Process

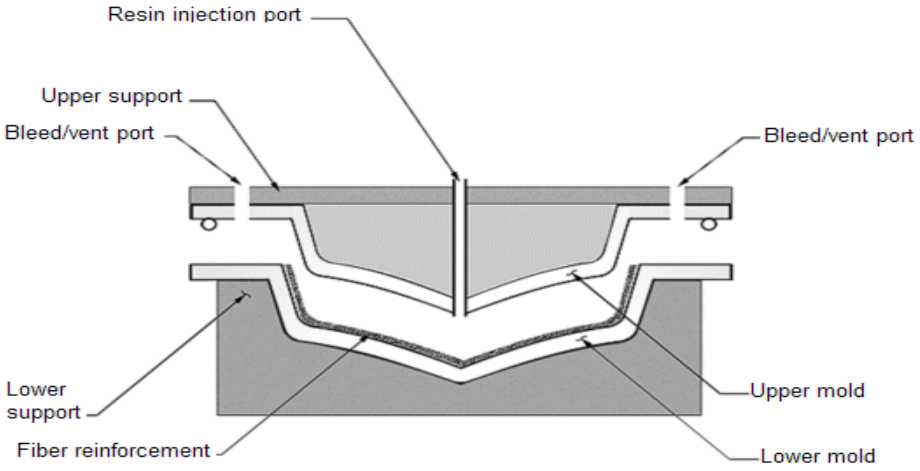


Figure 2.8Resin Transfer Molding

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Chapter 3

EXPERIENTAL STUDY OF WEAR CHARACTERISTICS OF RICE HUSK CHAR EPOXY COMPOSITE

CHAPTER – 3

3.1 INTRODUCTION:

Wear is probably the most important yet at least understood aspects of tribology. It is certainly the youngest of the tri of topics, friction, lubrication and wear, to attract scientific attention, although its practical significance has been recognizes throughout the ages. The findings of Guillaume Amontons in 1699 [70] establishing scientific studies of friction are almost of 300 years age, while Petrov [71], Tower [72] and Reynolds [73] brought enlightenment to the subject of lubrication a century ago in the hectic 1880s. Substantial Studies of wear can be associated only with the five decades that have elapsed since R. Holm [74] explored the fundamental aspects of surface interactions encountered in electrical contacts.

One third of our global energy consumption has been devoured wastefully in friction. In addition to the primary saving of energy, very significant additional economics can be made by the reduction of the cost involved in the manufacture and replacement of prematurely worn out components. The dissipation of energy by wear impairs strongly to the national economy and the life style of most of the peoples. So, the effective decrease and control of wear of metals are always desired [75].

Wear causes an enormous annual expenditure by industry and consumers. Most of this is replacing or repairing equipment that has worn to the extent that it no longer performs a useful function. For many machine components, this occurs after a very small percentage of the total volume has been worn away. For some industries, such as agriculture, as many as 40% of the components replaced on equipment have failed by abrasive wear. Other major sources of expenditure are losses production consequential upon lower efficiency and plant shutdown, the need to invest more frequently in capital equipment and increased energy consumption as equipment wears. Estimates of direct cost of abrasive wear to industrial nations vary from 1 to 4 % of gross national product and Rigney [76] has estimated that about 10% of all energy generated by man is dissipated in various friction processes.

Wear is not an intrinsic material property but characteristics of the engineering system which depend on load, speed, temperature, hardness, presence of foreign material and the environmental condition [77]. Widely varied wearing conditions causes wear of materials. It may be due to surface damage or removal of material from one or both of two solid surfaces in a sliding, rolling or impact motion relative to one another. In most cases wear occurs through surface interactions at asperities. During relative motion, material on contacting surface may be removed from a surface, may result in the transfer to the mating surface, or may break loose as a wear particle. The wear resistance of materials is related to its microstructure may take place during the wear process and hence, it seems that in wear research emphasis is placed on microstructure [78]. Wear of material depends on many variables, so wear research program must be planned systematically. Therefore researchers have normalized some of the data to make them more useful. The wear map proposed by Lim [79] is very much useful in this regard to understand the wear mechanism in different sliding conditions as well as the anticipated rates of wear.

3.2 RECENT TRENDS IN WEAR RESEARCH:

Numerous wear researches have been carried out in the 1940's and 1950's by mechanical engineers and metallurgists to generate data for the construction of motor drive, trains, brakes, bearings, bushings and other types of moving mechanical assemblies [80].

It became apparent during the survey that wear of materials was a prominent topic in a large number of the responses regarding some future priorities for research in tribology. Some 22 experienced technologists in this field, who attended the 1983 'Wear of Materials Conference' in Reston, prepared a ranking list [81]. Their proposals with top priority were further investigations of the mechanism of wear and this no doubt reflects the judgments that particular effects of wear should be studied against a background of the basic physical and chemical processes involved in surface interactions. The list proposed is shown in table 3.1.

Peterson [75] reviewed the development and use of tribo-materials and concluded that metals and their alloys are the most common engineering materials used in wear applications.

Grey cast iron for example has been used as early as 1388. Much of the wear research conducted over the past 50 years is in ceramics, polymers, composite materials and coatings [82].

Table-3.1 Priority in wears research [81]

Ranking	Topics
1.	Mechanism of Wear
2.	Surface Coatings and treatments
3.	Abrasive Wear
4.	Materials
5.	Ceramic Wear
6.	Metallic Wear
7.	Polymer Wear
8.	Wear with Lubrication
9.	Piston ring-cylinder liner Wear
10.	Corrosive Wear
11.	Wear in other Internal Combustion Machine component

Wear of materials encountered in industrial situations can be grouped into different categories as shown in table 3.2. Though there are situations where one type changes to another or where two or more mechanism plays together.

Table-3.2 Type of wear in industry [80]

Type of wear in Industry	Approximate percentage involved
Abrasive	50
Adhesive	15
Erosion	8
Fretting	8
Chemical	5

3.3 THEORY OF WEAR:

Wear occurs as a natural consequence when two surfaces with a relative motion interact with each other. Wear may be defined as the progressive loss of material from contacting surfaces in relative motion. Scientists have developed various wear theories in which the Physico-Mechanical characteristics of the materials and the physical conditions (e.g. the resistance of the rubbing body and the stress state at the contact area) are taken in to consideration. In 1940 Holm [83] starting from the atomic mechanism of wear, calculated the volume of substance worn over unit sliding path.

Barwell and Strang [84] in 1952: Archard [85] in 1953 and Archard and Hirst [86] in 1956 developed the adhesion theory of wear and proposed a theoretical equation identical in structure with Holm's equation. In 1957, Kragelski [87] developed the fatigue theory of wear. This theory of wear has been widely accepted by scientists in different countries. Because of the Asperities in real bodies, their interactions in sliding is discrete, and contact occurs at individual locations, which, taken together, form the real contact area. Under normal force the asperities penetrate into each other or are flattened out and in the region of real contact points corresponding stress and strain rise. In sliding, a fixed volume of material is subjected to the many times repeated action, which weakens the material and leads finally to rupture. In 1973, Fleischer [88] formulated his energy theory of wear. The main concept of this theory is that the separation of wear particles requires that a certain volume of material accumulates a specific, critical store of internal energy. It is known that a large part of the work done in sliding is dissipated as heat, and that small proportion of it accumulates in the material as internal potential energy. When the energy attains a critical value, plastic flow of the material occurs in this volume or a crack is formed. Further theories of wear are found in [87]. Though all the theories are based on different mechanisms of wear, the basic consideration is the frictional work.

In past few decades, numerous research works have been carried out on abrasive wear performance of polymer and polymer based composite in view of their extensive application in the field industry and agricultural sectors where abrasive wear is a predominant mode of failure. Conveyor aids, vanes, gears, bushes, seals, bearings, chute liners etc. are some examples of their applications [89-93].Since abrasive wear is the most severe form of wear accounting for 50% of

total wear, several researches has been devoted for exploring abrasive wear of polymer composites. Evans et al. [94] studied the abrasion wear behavior for 18 polymers and they noticed that low density polyethylene (LDPE) showed the lowest wear rate in abrasion against rough mild steel, but a higher wear rate in abrasion with coarse corundum paper. Unal et al. [95] studied abrasive wear behavior of polymeric materials. They concluded that the specific wear rate decreases with the decrease in abrasive surface roughness. They also concluded that, the abrasive wear include micro-cracking, micro-cutting, and micro-ploughing mechanisms. Whereas in another investigation [96] they concluded that the sliding speed has stronger effect on the specific wear rate. Shipway and Ngao [97] investigated the abrasive behavior of polymeric materials in micro-scale level. They concluded that the wear behaviour and wear rates of polymers depended critically on the polymer type. Harsha and Tewari [98]investigated the abrasive wear behaviour of polyaryletherketone (PAEK) and its composites against SiC abrasive paper. They concluded that the sliding distance, load, abrasive grit size have a significant influence on abrasive wear performance. Further there are many references that illustrate the influence of fillers and fiber reinforcement on the abrasive wear resistance of polymeric composites. Cirino et al. [91,100] investigated the sliding and abrasive wear behavior of polyetheretherketone (PEEK) with different continuous fiber types and reported that the wear rate decreases with increase in the fiber content. Chand et al. [101] studied low stress abrasive wear behavior of short E-glass fiber reinforced polymer composites with and without fillers by using rubber wheel abrasion test apparatus. They reported that higher weight fraction of glass fibers (45%) in the composites improves the wear resistance as compared to the composite containing less glass fibers (40%). Bijwe et al. [102] tested polyamide 6, polytetrafluoroethylene (PTFE) and their various composites in abrasive wear under dry and multi-pass conditions against silicon carbide (SiC) paper on pin-on-disc arrangement. They concluded that the polymers without fillers had better abrasive wear resistance than their composites. Liu et al. [103]investigated the abrasive wear behavior of ultrahigh molecular weight polyethylene (UHMWPE) polymer. They concluded that the applied load is the main parameter and the wear resistance improvement of filler reinforced UHMWPE was attributed to the combination of hard particles which prevent the formation of deep, wide and continuous furrows.

With regards to the usage of natural fiber as reinforcement for tribological application in polymeric composite, few works have been attempted. However, in recent years, some work has

been done on natural fiber like jute [104], cotton [105,106], sugarcane [107], oil palm [108], coir [109], kenaf [110], betel-nut [111], betel palm [112], wood flour [113] and bamboo powder [114] as reinforcement. In these works, the wear resistance of polymeric composites has been improved when natural fiber introduced as reinforcement.

3.4 TYPES OF WEAR:

In most basic wear studies where the problems of wear have been a primary concern, the so-called dry friction has been investigated to avoid the influences of fluid lubricants.

Dry friction is defined as friction under not intentionally lubricated conditions but it is well known that it is friction under lubrication by atmospheric gases, especially by oxygen [115].

A fundamental scheme to classify wear was first outlined by Burwell and Strang [116]. Later Burwell [117] modified the classification to include five distinct types of wear, namely (1) Abrasive (2) Adhesive (3) Erosive (4) Surface fatigue (5) Corrosive.

3.4.1 Abrasive wear:

Abrasive wear can be defined as wear that occurs when a hard surface slides against and cuts groove from a softer surface. It can be account for most failures in practice. Hard particles or asperities that cut or groove one of the rubbing surfaces produce abrasive wear. This hard material may be originated from one of the two rubbing surfaces. In sliding mechanisms, abrasion can arise from the existing asperities on one surface (if it is harder than the other), from the generation of wear fragments which are repeatedly deformed and hence get work hardened for oxidized until they became harder than either or both of the sliding surfaces, or from the adventitious entry of hard particles, such as dirt from outside the system. Two body abrasive wear occurs when one surface (usually harder than the second) cuts material away from the second, although this mechanism very often changes to three body abrasion as the wear debris then acts as an abrasive between the two surfaces. Abrasives can act as in grinding where the abrasive is fixed relative to one surface or as in lapping where the abrasive tumbles producing a

series of indentations as opposed to a scratch. According to the recent tribological survey, abrasive wear is responsible for the largest amount of material loss in industrial practice [118].

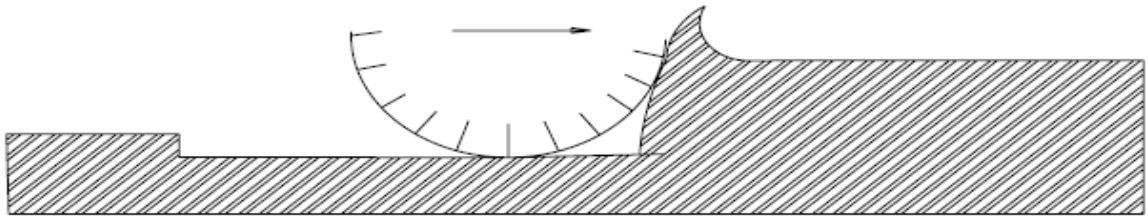

Figure-3.1 Schematic representations of the abrasion wear mechanism

3.4.2 Adhesive wear:

Adhesive wear can be defined as wear due to localized bonding between contacting solid surfaces leading to material transfer between the two surfaces or the loss from either surface. For adhesive wear to occur it is necessary for the surfaces to be in intimate contact with each other. Surfaces, which are held apart by lubricating films, oxide films etc. reduce the tendency for adhesion to occur.

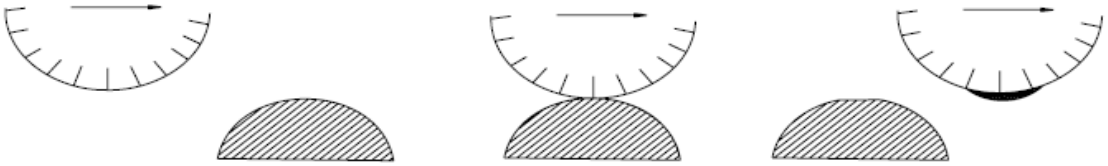

Figure-3.2 Schematic representations of the adhesive wear mechanism

3.4.3 Erosive wear:

Erosive wear can be defined as the process of metal removal due to impingement of solid particles on a surface. Erosion is caused by a gas or a liquid, which may or may not carry, entrained solid particles, impinging on a surface. When the angle of impingement is small, the

wear produced is closely analogous to abrasion. When the angle of impingement is normal to the surface, material is displaced by plastic flow or is dislodged by brittle failure.

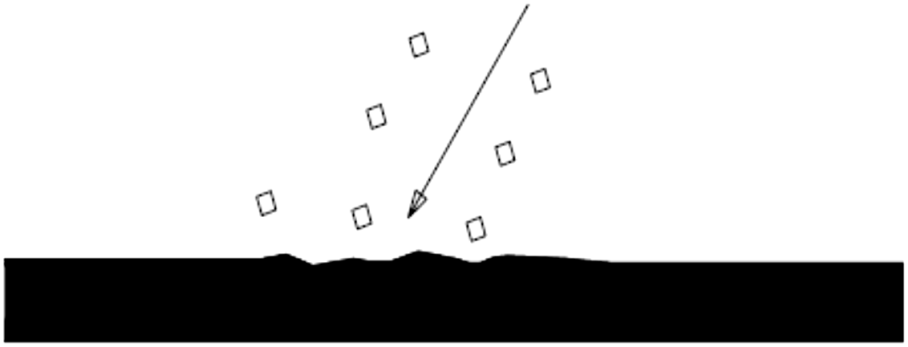

Figure-3.3 Schematic representations of the erosive wear mechanism

3.4.4 Surface fatigue wear:

Wear of a solid surface caused by fracture arising from material fatigue. The term 'fatigue' is broadly applied to the failure phenomenon where a solid is subjected to cyclic loading involving tension and compression above a certain critical stress. Repeated loading causes the generation of micro cracks, usually below the surface, at the site of a pre-existing point of weakness. On subsequent loading and unloading, the micro crack propagates. Once the crack reaches the critical size, it changes its direction to emerge at the surface, and thus flat sheet like particles is detached during wearing. The number of stress cycles required to cause such failure decreases as the corresponding magnitude of stress increases. Vibration is a common cause of fatigue wear.

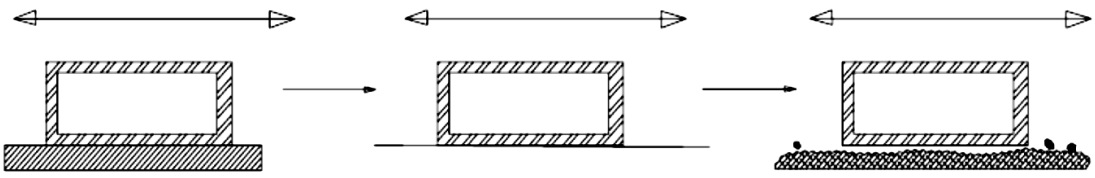

Figure-3.4 Schematic representations of the surface fatigue wear mechanism

3.4.5 Corrosive wear:

Most metals are thermodynamically unstable in air and react with oxygen to form an oxide, which usually develop layer or scales on the surface of metal or alloys when their interfacial bonds are poor. Corrosion wear is the gradual eating away or deterioration of unprotected metal surfaces by the effects of the atmosphere, acids, gases, alkalis, etc. This type of wear creates pits and perforations and may eventually dissolve metal parts.

3.5 SYMPTOMS OF WEAR:

A summary of the appearance and symptoms of different wear mechanism is indicated in Table-4.3 and the same is a systematic approach to diagnose the wear mechanisms.

Table-3.3 Symptoms and appearance of different types of wear

Types of wear	Symptoms	Appearance of the worn-out surface
Abrasive	Presence of clean furrows cut out by abrasive particles.	Grooves
Adhesive	Metal transfer is the prime symptoms.	Seizure, catering rough and torn-out surfaces.
Erosion	Presence of abrasives in the fast moving fluid and short abrasion furrows.	Waves and troughs.
Corrosion	Presence of metal corrosion products.	Rough pits or depressions.
Fatigue	Presence of surface or subsurface cracks accompanied by pits and spalls.	Sharp and angular edges around pits.
Impacts	Surface fatigue, Small sub-micron particles or formation of spalls.	Fragmentation, peeling and pitting.
Delamination	Presence of subsurface cracks parallel to the surface with semi-dislodged or loose flakes.	Loose, long and thin sheet like particles

Literature available on the rate of controlling abrasive wear mechanism demonstrate that it may change abruptly from one another at certain sliding velocities and contact loads, resulting in abrupt increases in wear rates. The conflicting results in the abrasive wear literature arise partly because of the differences in testing conditions, but they also make clear that a deeper understanding of the abrasive wear mechanism is required if an improvement in the wear resistances of the polymer matrix composites is to be achieved. This in turn requires a systematic study of the wear under different loads and velocities. It is generally recognized that abrasive wear is a characteristic of a system and influenced by many parameters. Laboratory scale investigation if designed properly allows careful control of the tribo system where by the effects of different variables on wear behavior of PMCs can be isolated and determined. The data generated through such investigation under controlled conditions may help in correct interpretation of the results.

As new developments are still under way to explore innovative fields for tribo-application of natural fiber base materials, in this chapter an attempt has been made to study the potential of using Rice husk ceramic fiber (RHC) for tribological applications. In the current study the effect of fiber loading, sliding velocity and normal load on abrasive wear behaviour of carburized rice husk ceramic field epoxy composite has been evaluated and possible wear mechanism has been discussed with SEM observation.

3.6 RAW MATERIALS USED:

Raw materials used in this experimental work are listed below:

1. Natural fiber (rice husk)
2. Epoxy resin
3. Hardener

3.6.1 Rice husk:

Rice husk (RH) is an agricultural waste material abundantly available in rice-producing countries. They are the natural sheaths that from on rice grains during their growth. Removal during the refining of rice, these husks have no commercial interest [7]. The annual rice husk production in India amounts is generally approximately 12 million tons. Rice husk is generally not recommended as cattle feed since cellulose and other sugar contents are low. Worldwide

production of rice husk is about 120 million tons per year. That makes the rice husk one of the largest readily available but also one of the most under-utilized resources. Increase of environmental awareness has led to a growing interest in researching ways of an effective utilization of rice by-product, from which rice husk is particularly valuable due to its high content of amorphous silica [119,120]. The rice husk contains 80 percent organic volatile materials and remaining 20 percent silica. The rice husk ash (RHA) contains 85 percent to 95 percent amorphous silica. the chemical composition of the rice husk ash varies from sample to sample which may be due to the different geographical conditions, type of paddy, climatic conditions and type of fertilizer used [8].

It was found that RH char contains amorphous silica in addition to amorphous carbon as the main constituents [120]. Compares to the conventional sources of silica rice husk ash as a char raw material possess advantages like fine particles size and higher reactivity due to its amorphous nature. Many authors have concluded that rice husk is an excellent source of high grade amorphous silica [28-31]. Accordingly silica is used in different industries and products like rubber industry as a reinforcing agent, in tooth pastes as a cleaning agent, as an anti-caking agent in salts, in cosmetics etc.

But it is interesting to note that rice husk contains 20% ash, 22% lignin, 38% cellulose, 18% pentosans and 2% moisture. Silica obtained from rice husk ash is highly reactive, depending upon the degree of firing and is used for making insulating materials, refractory bricks, Portland cement, masoriry cement and pottery ware. It is used as filler materials in paints and in fertilizers etc. rice husk ash has also been used in aluminum alloy for increasing abrasion resistance [32].

In the present work, volume fractions of rice husk char fibers (10%, 20%, 30% and 40% by weight) have been taken as reinforcement in the polymer matrix.

3.6.2 Epoxy resin:

Epoxy is a copolymer; that is, it is formed from two different chemicals. These are referred to as the "resin" and the "hardener". The resin consists of monomers or short chain polymers with an epoxide group at either end. Most common epoxy resins are produced from a reaction between -epichlorohydrin and bisphenol-A, though the latter may be replaced by similar

chemicals. The hardener consists of polyamine monomers, for example triethylenetetramine (TETA). When these compounds are mixed together, the amine groups react with the epoxide groups to form a covalent bond. Each NH group can react with an epoxide group, so that the resulting polymer is heavily cross linked, and is thus rigid and strong.

Softener (Araldite LY 556) made by CIBA GEIGY limited having the following outstanding properties has been used as the matrix material.

a. Excellent adhesion to different materials.
b. High resistance to chemical and atmospheric attack.
c. High dimensional stability.
d. Free from internal stresses.
e. Excellent mechanical and electrical properties.
f. Odorless, tasteless and completely nontoxic.
g. Negligible shrinkage.

3.6.3 Hardener:

Hardener (HY951), aliphatic primary amines which has a viscosity of 10-20 MPa at 25ºc is used along with matrix material.

3.7 EXPERIEMENT

3.7.1 Preparation of RH Char:

Rice husk obtained from mills directly were cleaned, sheared from dusts and kept in a oven for one day at the temperature of 110 ^{0}c. They were sieved to a size of 100 μm**.** They were carbonized in absence of atmospheric gas. The carburization temperature selected was 850˚c, 900°c and 950˚c. The required quantity rice husk were taken in crucibles were placed in the furnace. It took about one and half hours to reach the required temperature. at this temperature one hour soaking time was allowed . After this 24hrs cooling was allowed so that the furnace comes to room temperature. Then the carburized rice husks were taken out from the furnace for further use. Figure 3.5 Shows the different stages in the preparation of RH-char.

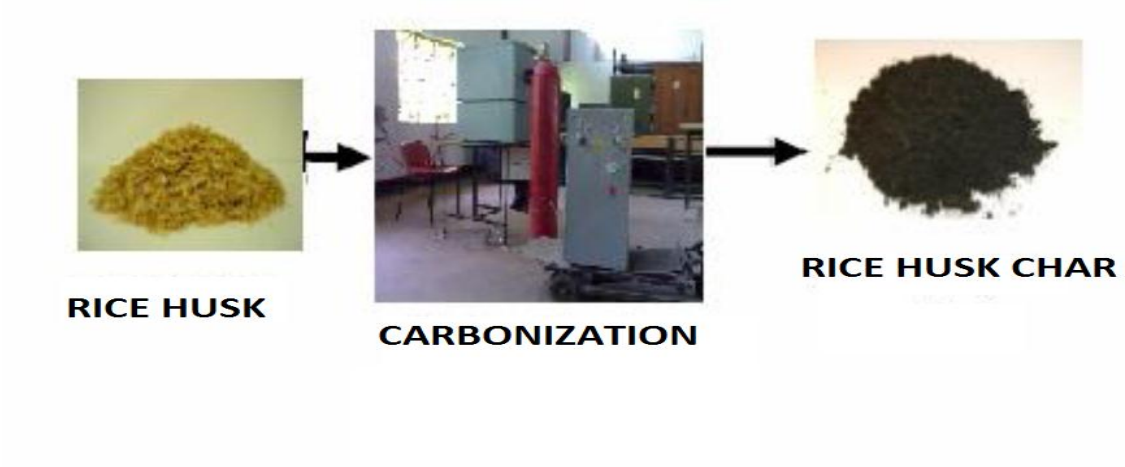

Figure-3.5 Preparation of RH-Char

3.7.2 Preparation for the test specimens:

The different amount of fibers has been added to the resin to prepared composite samples with 10, 20, 30 and 40% volume fraction of fiber. To manufacture pin type composite sample a steel mould has been used in this work which is shown in Figure 3.6. The mixture of carburized rice husk char fiber and resin has been poured into the cylindrical cavity present in the mould and then the two halves of the mould fixed properly. During fixing some of the resin mix has been squeezed out. Therefore care has been taken for this in the experiment to make composite pins of length 35 mm and diameter of 10 mm. The samples were kept in the moulds for curing at room temperature (28 ^{0}C) for 24 hr. For the purpose of comparison the matrix material was also cast under similar condition. After curing the samples were taken out from the mould, finished ground to required shape, sizes for wear testing.

3.7.3 Measurement of Density and Voids content:

The density and the void content of composite sample have been determined as per ASTM-C 639 and ASTM D-2734-70 standard procedure respectively. The volume fraction of voids (Vv) in the composites was calculated by using equation:

$$V_v = \frac{\rho_t - \rho_a}{\rho_t} \qquad (3.1)$$

Where ρ_t and ρ_a are the theoretical and actual density of composite respectively.

3.7.4 Dry sliding wear test:

Dry sliding wear test has been carried out under multi-pass condition on a pin-on-disc type wear testing machine (As per ASTM G-99 standard) supplied by Magnum, Bangalore (Figure-3.7). Abrasive paper of 400 grade (grit-23 µm) has been pasted on a rotating disc (EN 32 Steel disc) of 120mm diameter using double-sided adhesive tape. The specimens under tests were fixed to the sample holder. The holder along with the specimen (Pin) was positioned at a particular track diameter. This track diameter is to be changed after each test (i.e.) a fresh track is to be selected for each specimen. A track radius of 40mm was selected for this experiment and was kept constant for the entire investigation. For each test new abrasive paper was used and the sample was abraded for a total sliding distance of 189.9 m. During experiment the specimen remains fixed and disc rotates. Load is applied through a dead weight loading system to press the pin against the disc. The speed of the disc or motor rpm can be varied through the controller and interval of time can be set by the help of timer provided at the control panel. The test conditions under which the experiment has been conducted are presented in Table 3.5. The mass loss in the specimen after each test was estimated by measuring the weight of the specimen before and after each test using an electronic balance with an accuracy of ± 0.001 mg. Care has been taken that the specimen under test are continuously cleaned with woolen cloth to avoid entrapment of wear debris and to achieve uniformity in the experimental procedure. Test pieces are also cleaned with acetone prior and after each test. The machine is fixed with data acquisition system with 'MAGVIEW-2007' software from which the frictional force that arises at the contact can be read out/recorded directly. For a particular type of composite 5 sets of test pieces were tested.

3.7.5 Calculation for Wear:

Wear rate was estimated by measuring the weight loss of the specimen after each test. The weight loss was calculated by taking the weight difference of the sample before and after each test. The weight loss:

$$(\Delta w) = (w_a - w_b)\,\text{gm} \qquad (3.2)$$

Where Δw is the weight loss in gm and w_a and w_b are the weight of the sample after and before the abrasion test in gm. The abrasive wear rate (*W*) can be calculated by using the following formula:

$$\mathbf{W_r} = \Delta w / \mathbf{L} \qquad (3.3)$$

where *'W'* is the wear rate in N/m, Δ*w the* weight loss in gm. and '*L'* is the sliding distance in m. The average value of weight loss and wear rate for each batch is listed in Table-3.6 to 3.9.

The volumetric wear rate $W_{v\ (m^3/sec.)}$ of the composite is relate to density (ρ) and the abrading time (t), was calculated using the expression,

$$\mathbf{W_v} = \Delta w / \boldsymbol{\rho}\ \mathbf{t} \qquad (3.4)$$

For characterization of the abrasive wear behaviour of composite, the specific wear rate is employed. This is defined as the volume loss of the composite per unit sliding distance and per unit applied load. Often the inverse of the specific wear rate can be expressed in terms of volumetric wear rate. The specific wear rate (k_0) can also be calculated by using equation:

$$\mathbf{W_s} = \mathbf{W_v} / \mathbf{V_s}\ \mathbf{F_n} \qquad (3.5)$$

where 'w_s' is the specific wear rate in m^3/Nm, 'Δw' is the weight loss in grams, 'V_s' is the sliding velocity in meter per second, and '*Fn'* is the applied load in N.

Experimental results of the wear test of different test pieces (10, 20, 30 and 40% by weight of Rice husk char) at different test conditions are tabulated and presented in table .3.6-3.9

3.8 RESULTS AND DISCUSSION:

Based on the tabulated results, various graphs are plotted and presented in figs 3.6-3.9 for different percentages of reinforcement under different test conditions.

Fig 3.8-3.19 shows the variation of wear rate with sliding distance for different loads (5, 10, 15 and 20N) at sliding velocity of 0.633 $m.sec^{-1}$. It is seen from the plot that with addition of RHA char particles the wear rate of the composite decreases. it is also seen from the plots that the wear rate first decrease and then almost remains same for the entire test period. Since the trend for other sliding velocities remains same it has not been presented here.

Fig 3.20-3.22 show the variation of specific wear rate with filler volume fraction (i.e.) RH char, it is seen from the plot that specific wear rate of the composite decreases with increase in fiber volume fraction. This also shows that irrespective of the load the nature of the curves remains same only the values of the respective specific wear decreases, thus it can be concludes that addition of RH char is beneficial in improving the wear resistance of pure epoxy.

Fig 3.23-3.25 25shows the specific wear rate ($\mathbf{w_s}$) at 15 N applied load in abrasive wear mode as a function of sliding distance for all the composites. The specific wear rate decreases with increasing sliding distance for all the samples. Initial, maximum wear rate was observed because abrasive paper was fresh. With consecutive runs wear rate decreases gradually because the abrasive grits become smooth and less effective. The wear debris filled the space between the abrasives, which reduces the depth of penetration in the sample. The composite with 30 wt. % reinforcement showed the minimum wear rate while the composite with 10 vol. % reinforcement showed the maximum wear rate. This attributed to the fact that in abrasive wear mode the wear debris consisting of mainly RH char particles and found maximum for 10wt % composite and minimum for 30 wt % composite

Fig 3.26-3.28 shows the influence of normal load on the abrasive wear behavior of reinforced composites for different volume fraction. It is observed that the wear rate of all composite sample increases with normal load. This is because at higher load frictional thrust increases; which results in increased debonding and fracture. A similar effect of normal load on volumetric wear rate has been observed by Cirinoet,al, [121] in the case of carbon epoxy composite, Verma et, al, [122] for GRP composite. Hence it can be conclude that, the abrasive

wear of RHC epoxy composite is very much sensitive to the normal load than the sliding velocity. The wear rate decreases with addition of RH char fibers up to30 wt %. It means that RH char is very effective in improving the tribological performance of epoxy, especially for its wear resistance.

Fig 3.29-3.31 Variation of wear rate with volume fraction at different normal load for different carbonization tempreture**.**

Fig 3.32-3.34 shows the variation of co-efficient of friction with respect to time. It is clear from the figure that the co-efficient of friction increases initially to a higher value due to the fresh abrasive paper and as the process continues it almost remains same for the entire test period. It is also seen that the co-efficient of friction decrease when the volume fraction of reinforcement is more.

3.9 WORN SURFACE MORPHOLOGY:

The worn surface morphologies of composites have been examined by scanning electron microscopy (SEM). In 30% reinforcement carbonized at 850˚c[Figure-3.30 (a)] shows crack lines are clearly visible, but are not so severe that the particle are detached from the matrix.. In 30% reinforcement carbonized at 900˚c [Figure-3.30 (b)] load it can be seen that Rough Surface with many tearing and branching. This tearing seems to be originate from the interface between matrix and filler. In 30% reinforcement carbonized at 950˚c [Figure-3.30 (c)] Some particles are detached from the body causing micro voids. Many micro cracks are observed to have originated from the region around the carbonized particles in the matrix.

This implies that carbonized material acted as stress concentrator and promoted the formation of cracks, when sample was loaded. It is possible that increasing filler loading, it increases wear properties of neat epoxy. As a conclusion 30% volume fraction of reinforcement and 950˚c carburized temperature given the best wear performance.

3.11 CONCLUSIONS:

Based on experimental results of abrasive wear of Rice husk char epoxy composite tested under different normal loads, sliding velocity and sliding distances, the following conclusions may be drawn:

- The Rice husk one of the byproduct of rice mill can successfully be utilized to produce composite by suitably bonding with resin for value added product.

- By carburizing the Rice husk, amorphous silica and carbon are obtained which provides the higher hardness values.

- The incorporation of Rice husk char into epoxy can significantly reduce abrasive wear loss. The optimum wear resistance property was obtained at the fiber content of 30% weight fraction.

- The specific wear rate of composite decreases with the increases of sliding distance because the space between the abrasive filled by the debris, which reduced the depth of penetration abrasive particle in to the composite sample.

- Friction coefficient of the composites decreases with addition of Rice husk char.

- Minimum wear rate is observed for 30% volume of reinforcement at 950˚c carburized temperature with 15N load.

Table-3.4 Density of neat epoxy and RHC reinforced composite samples

Fiber content (%)	Measured Density (gm/cm^3)
0	1.080
10	1.213
20	1.211
30	1.233
40	1.215

Table-3.5 Test parameter for Dry Sliding wear test

Test Parametes	Units	Values
Reinforcement Preparing Tempreture	$^{\circ}$c	850,900,950
Load (L)	N	5, 10, 15,and 20
Sliding Velocity (v)	m/s	0.63 (interval of time is 5min)
Track radius (r)	mm	40
Temperature	^{0}C	25

Table-3.6 Weight loss (Δw), Wear rate (*W*) , Specific wear rate (W_s) and volumetric wear rate (Wv) of tested composite samples made at 850°c for Sliding velocity =0.63m/s, Sliding distance =189.90m

Fiber content (vol %)	Load (N)	(Δw) (gm)	$W \times 10^{-5}$ (N/m)	$Ws \times 10^{-9}$ (m^3/N.m)	$Wv \times 10^{-9}$ (m^3/Sec.)
10	5	0.35	0.3641	0.061	0.192
	10	0.87	0.943	0.076	0.478
	15	1.34	1.4	0.078	0.736
	20	1.7	1.76	0.074	0.934
20	5	0.18	0.187	0.031	0.099
	10	0.63	0.655	0.055	0.346
	15	0.92	0.981	0.0537	0.506
	20	1.32	1.37	0.0578	0.726
30	5	0.29	0.301	0.049	0.156
	10	0.42	0.436	0.036	0.227
	15	0.86	0.894	0.049	0.464
	20	1.12	1.16	0.04	0.605
40	5	0.21	0.218	0.036	0.115
	10	0.39	0.405	0.03	0.213
	15	0.85	0.884	0.049	0.466
	20	1.04	1.08	0.045	0.57

Table-3.7 Weight loss (Δw), Wear rate (*W*) , Specific wear rate (W_s) and Volumetric wear rate(W_v)of tested composite samples made at 900°c for Sliding velocity =0.63m/s, Sliding distance =189.90m

Fiber content (vol %)	Load (N)	(Δw) (gm)	$W \times 10^{-5}$ (N/m)	$Ws \times 10^{-9}$ (m^3/N.m)	$Wv \times 10^{-9}$ (m^3/Sec.)
10	5	0.42	0.4369	0.073	0.23
	10	1.2	1.248	0.104	0.66
	15	1.73	1.799	0.1008	0.95
	20	2.18	2.267	0.095	1.198
20	5	0.24	0.2496	0.042	0.132
	10	0.54	0.5617	0.047	0.3
	15	1.01	1.05	0.059	0.556
	20	1.26	1.31	0.055	0.693
30	5	0.28	0.2913	0.048	0.151
	10	0.58	0.6033	0.05	0.313
	15	0.96	0.9986	0.055	0.52
	20	1.73	1.799	0.057	0.724
40	5	0.19	0.1976	0.033	0.104
	10	0.32	0.3329	0.028	0.175
	15	0.64	0.6658	0.037	0.351
	20	1.14	1.186	0.049	0.625

Table-3.8 Weight loss (Δw), Wear rate (*W*) , Specific wear rate (W_s) and volumetric wear rate (W_v) of tested composite samples made at 950°c for Sliding velocity =0.63m/s, Sliding distance =189.90m

Fiber content (vol %)	Load (N)	(Δw) (gm)	$W \times 10^{-5}$ (N/m)	$Ws \times 10^{-9}$ (m^3/N.m)	$Wv \times 10^{-9}$ (m^3/Sec.)
10	5	0.21	0.2184	0.036	0.115
	10	0.4	0.4161	0.034	0.219
	15	0.34	0.3537	0.019	0.186
	20	0.31	0.3225	0.0135	0.17
20	5	0.23	0.2392	0.04	0.126
	10	0.53	0.5513	0.046	0.292
	15	0.72	0.749	0.042	0.396
	20	0.98	1.0194	0.043	0.539
30	5	0.2	0.208	0.034	0.108
	10	0.32	0.3329	0.027	0.173
	15	0.54	0.5617	0.03	0.292
	20	0.83	0.8634	0.0357	0.448
40	5	0.19	0.1976	0.033	0.104
	10	0.26	0.2705	0.022	0.143
	15	0.38	0.3953	0.022	0.022

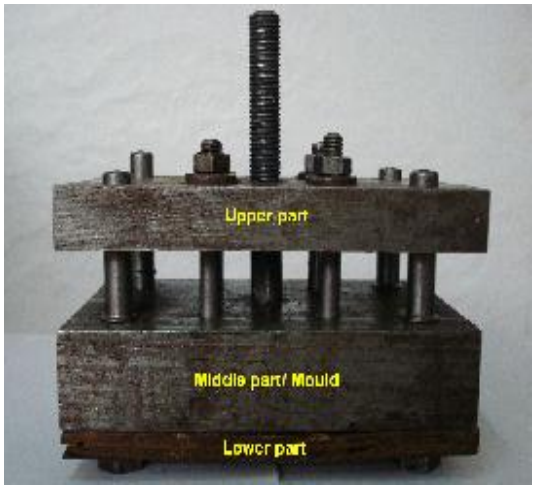

Figure-3.6(a)

Figure-3.6 (b)

Figure-3.6 (c)

Figure-3.6. Steel Mould and prepared pin type composite samples; (a) Mould used for preparing samples, (b) Two halves of the mould, (c) Fabricated Composite Pins

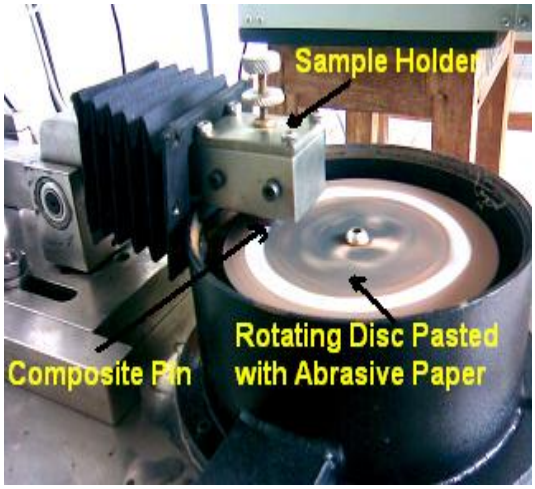

Figure-3.8 (a) **Figure-3.8 (b)**

Figure-3.7 Experimental set-up; (a) Pin-on-disc type wear testing machine, (b) Composite sample under abrasive wear test

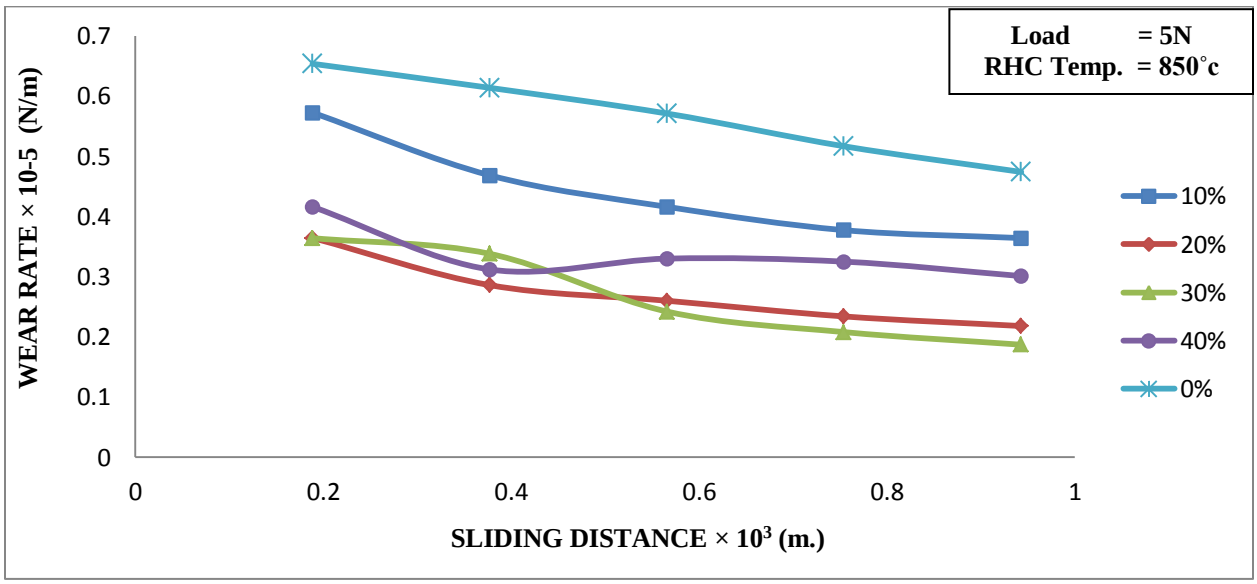

Figure-3.8 Variation of wear rate with sliding distance at load of 5N and carbonization temperature 850°c.

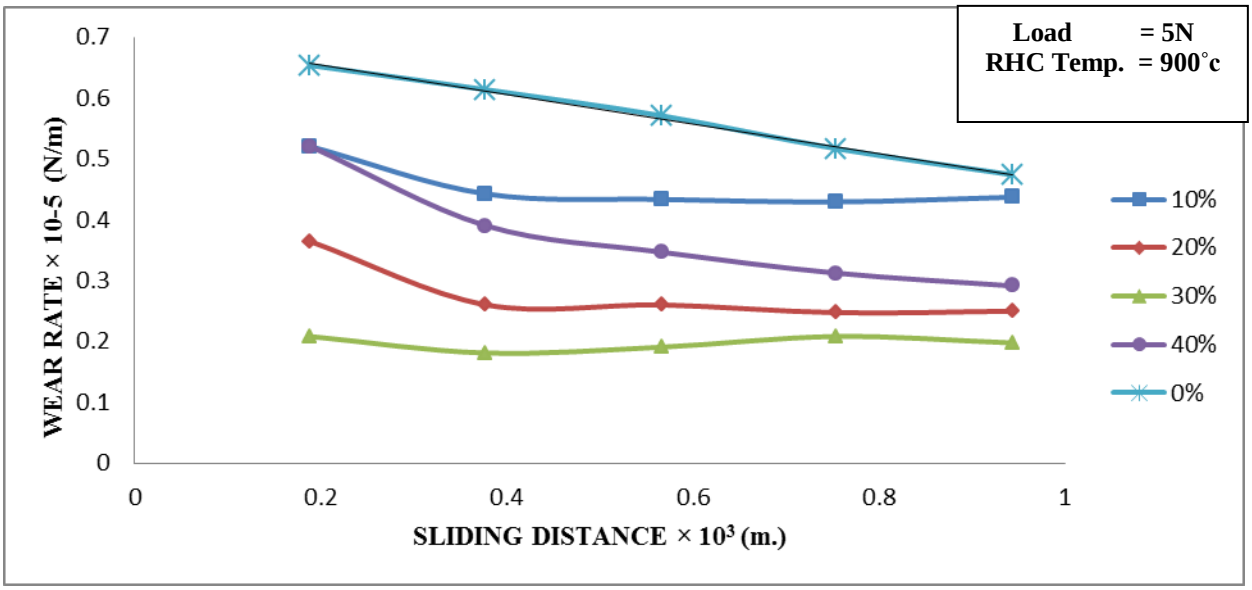

Figure-3.9 Variation of wear rate with sliding distance at load of 5N and carbonization temperature 900°c.

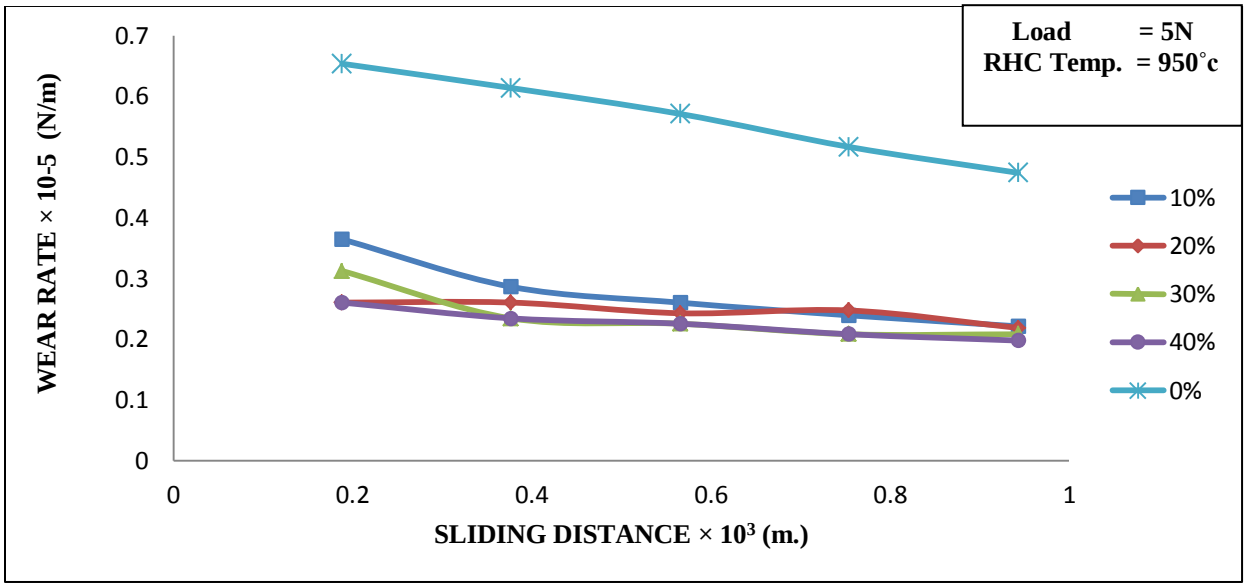

Figure-3.10 Variation of wear rate with sliding distance at load of 5N and carbonization temperature 950°c.

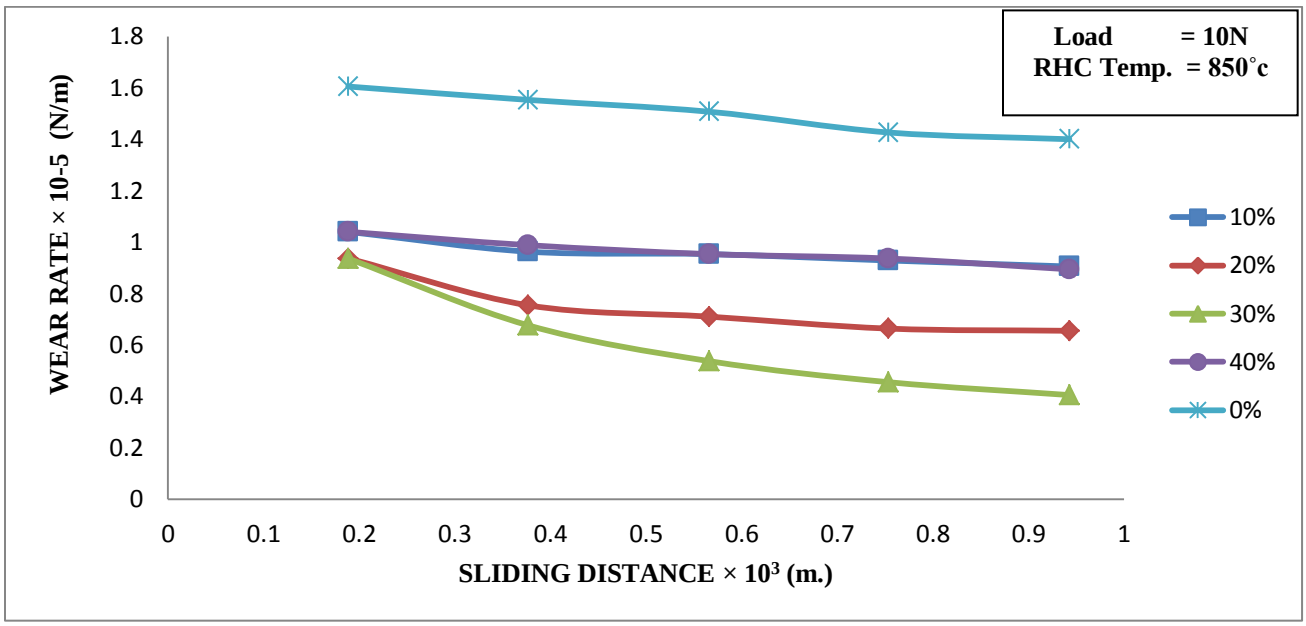

Figure-3.11 Variation of wear rate with sliding distance at load of 10N and carbonization temperature 850°c.

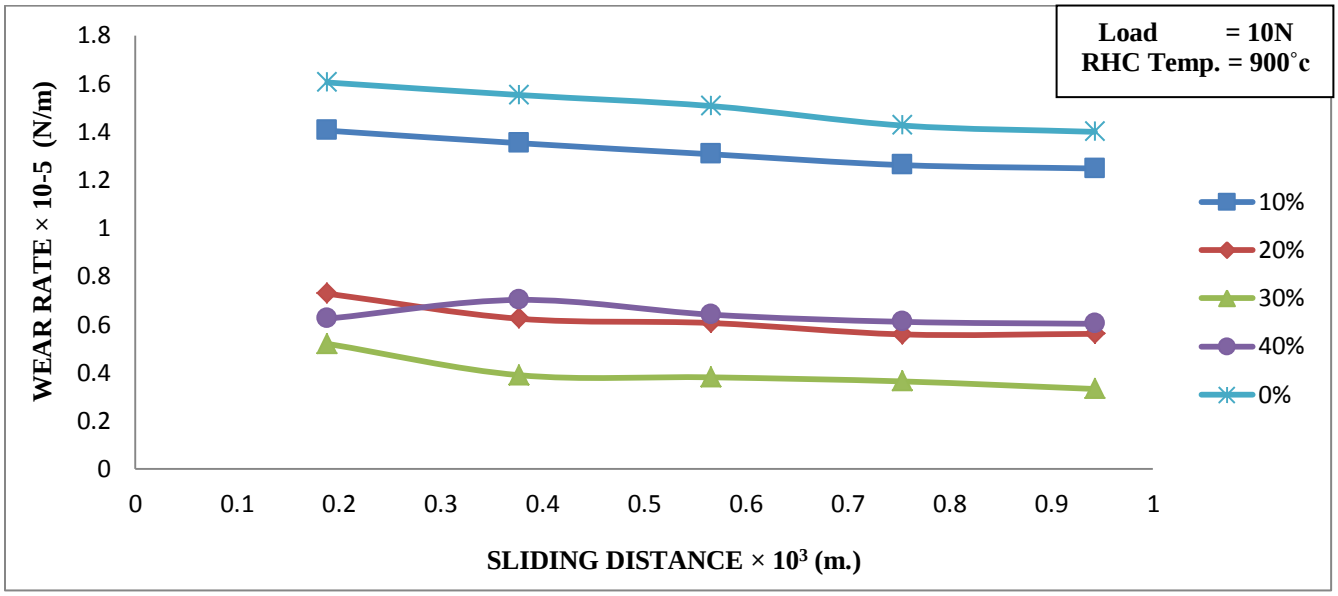

Figure-3.12 Variation of wear rate with sliding distance at load of 10N and carbonization temperature 900°c.

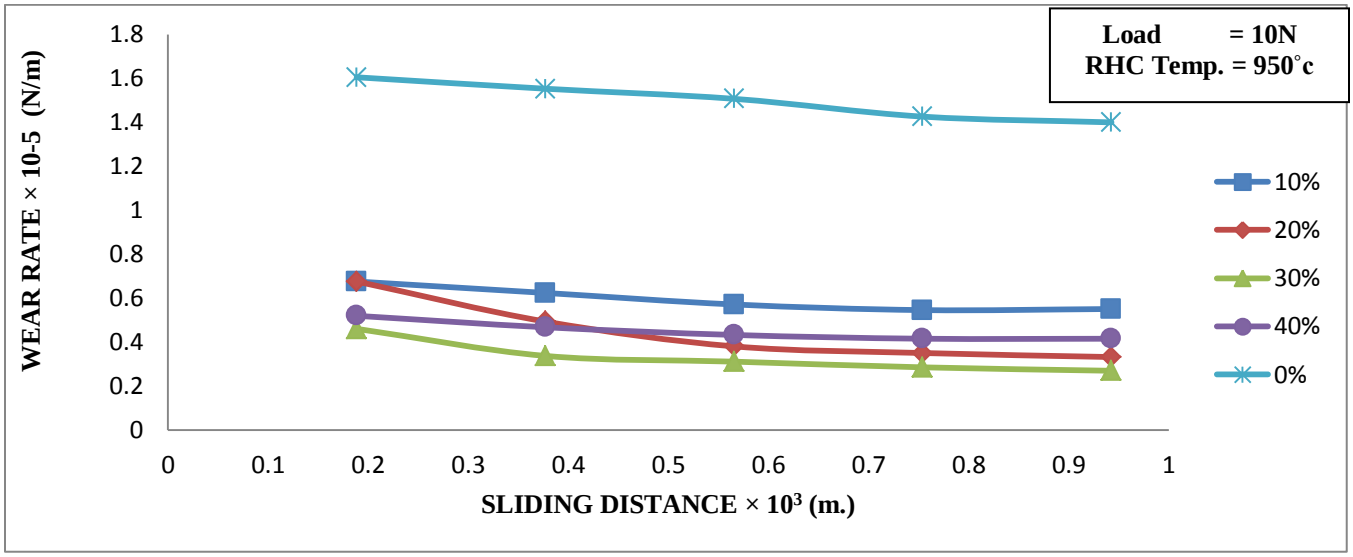

Figure-3.13 Variation of wear rate with sliding distance at load of 10N and carbonization temperature 950°c.

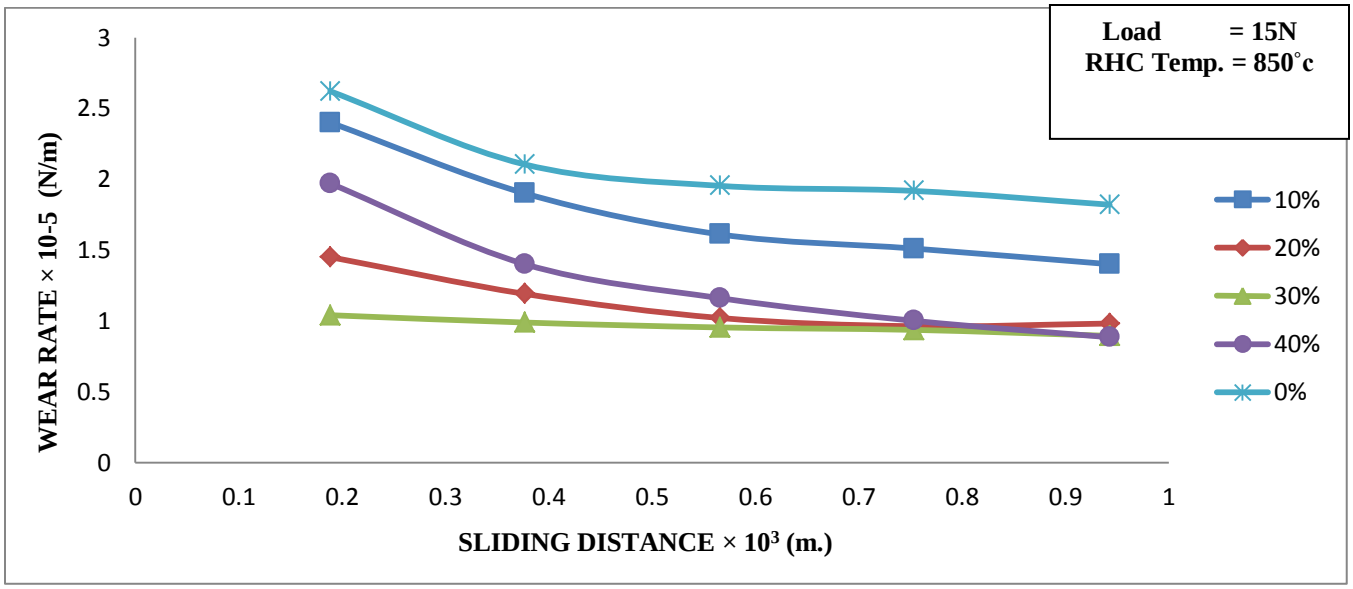

Figure-3.14 Variation of wear rate with sliding distance at load of 15N and carbonization temperature 850°c.

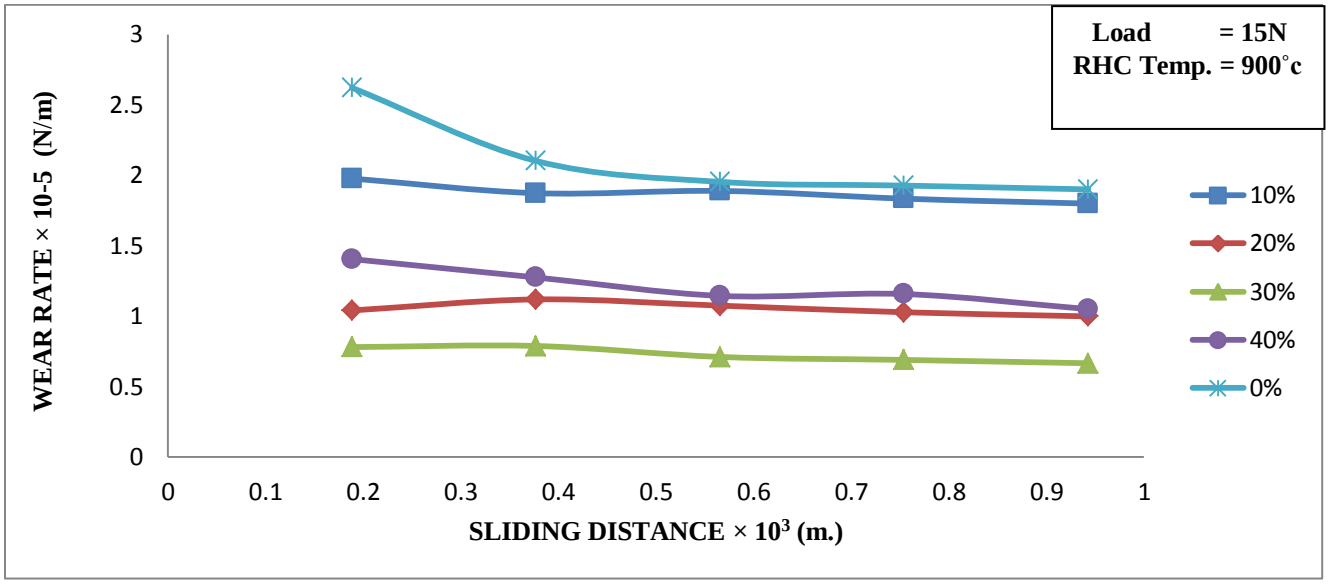

Figure-3.15 Variation of wear rate with sliding distance at load of 15N and carbonization temperature 900°c.

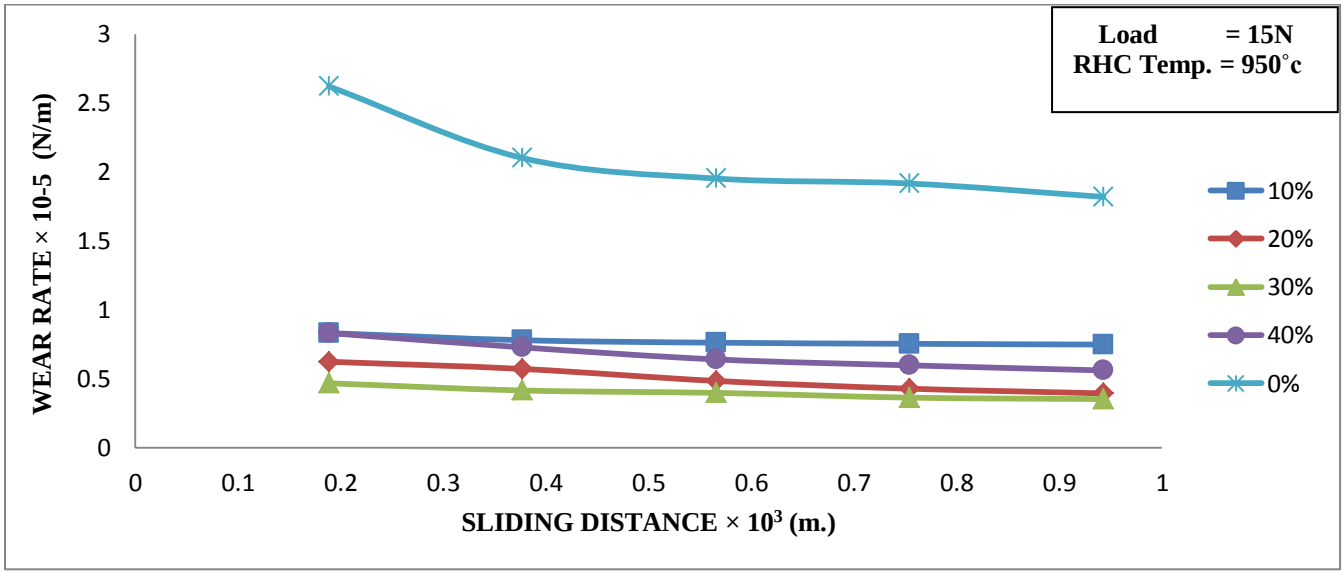

Figure-3.16 Variation of wear rate with sliding distance at load of 15N and carbonization temperature 950°c.

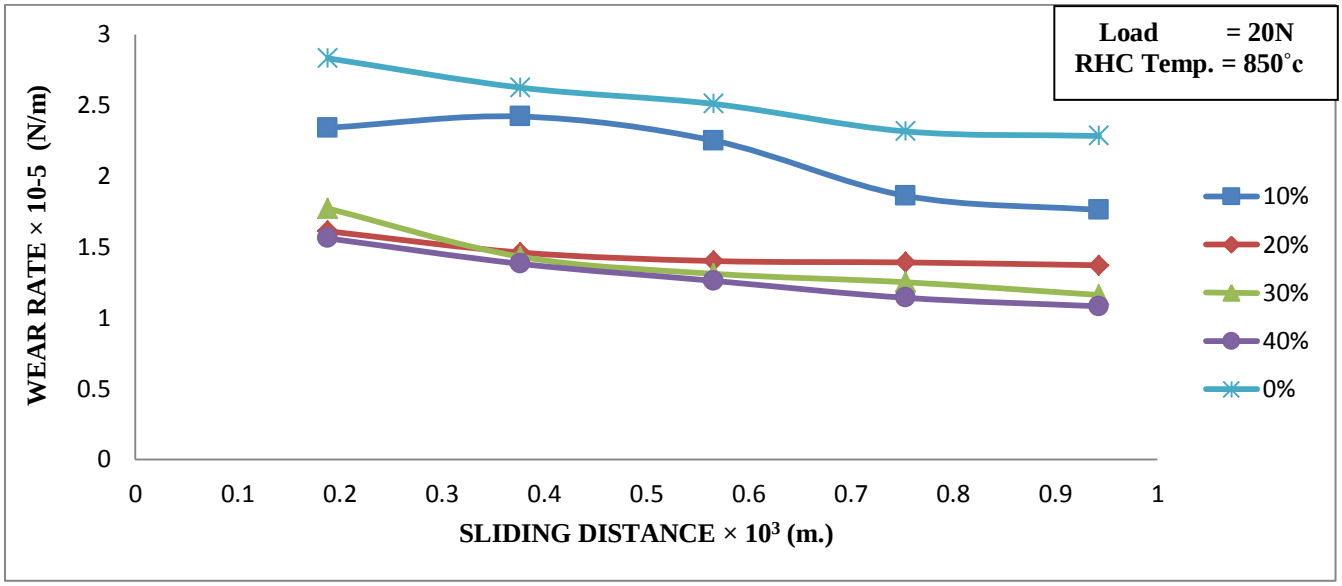

Figure-3.17 Variation of wear rate with sliding distance at load of 20N and carbonization temperature 850°c.

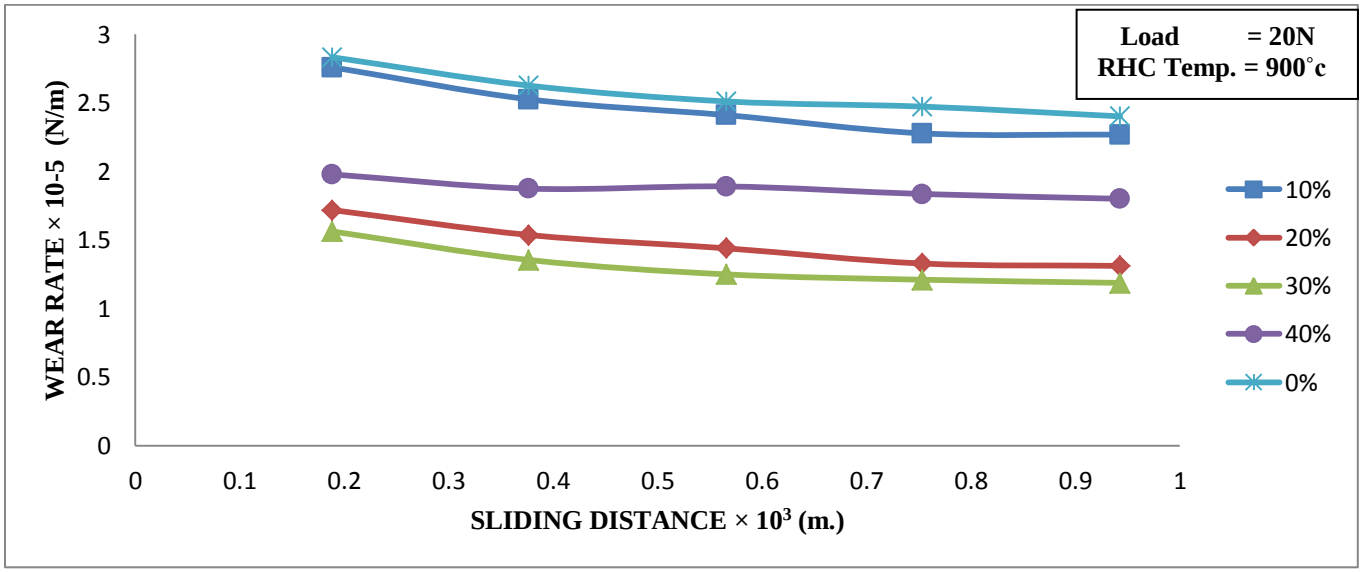

Figure-3.18 Variation of wear rate with sliding distance at load of 20N and carbonization temperature 900°c.

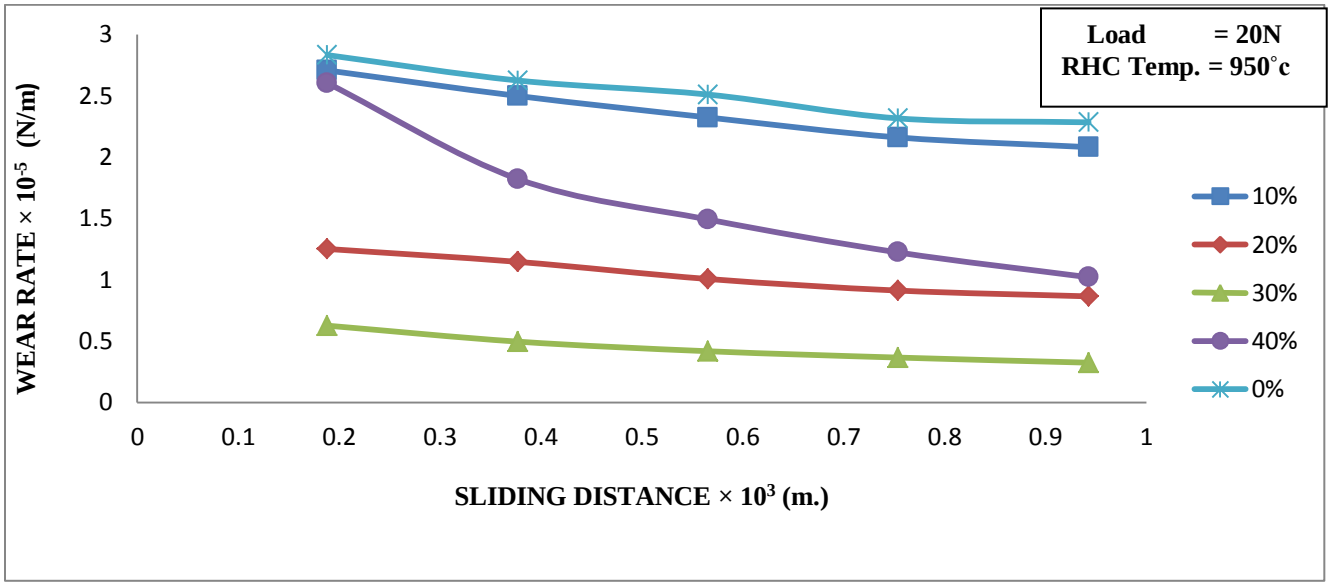

Figure-3.19 Variation of wear rate with sliding distance at load of 20N and carbonization temperature 950°c.

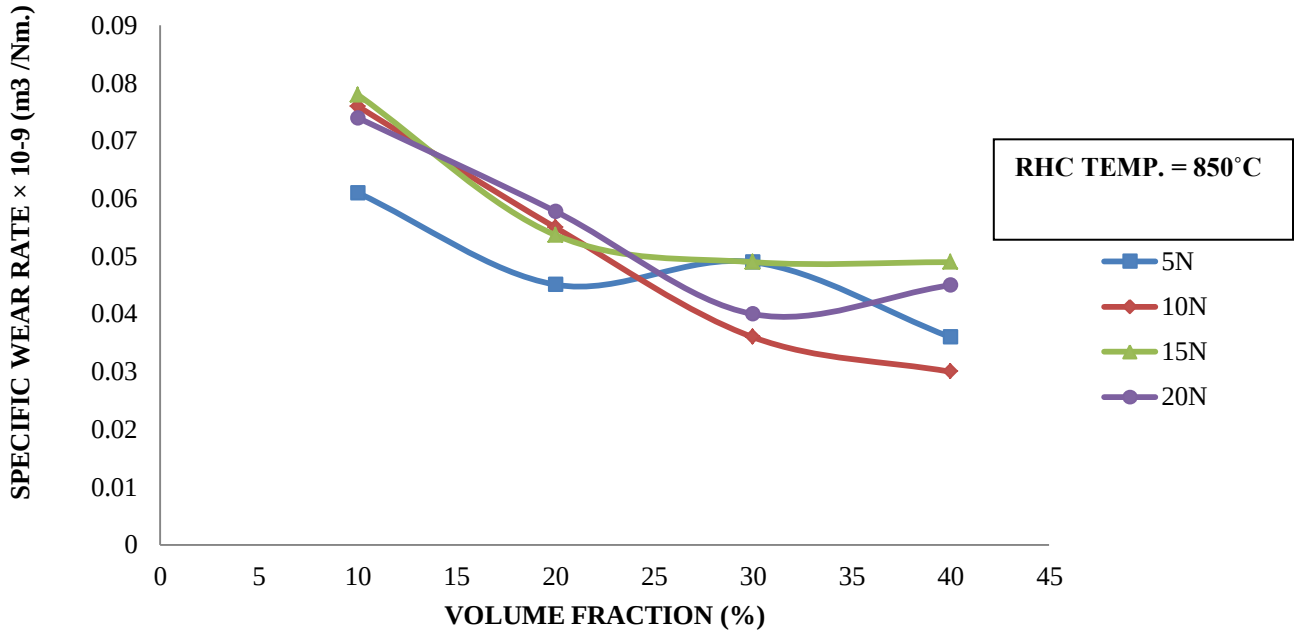

Figure-3.20 Variation of specific wear rate with different volume fraction of reinforcement at different normal load, carbonization temperature 850˚c.

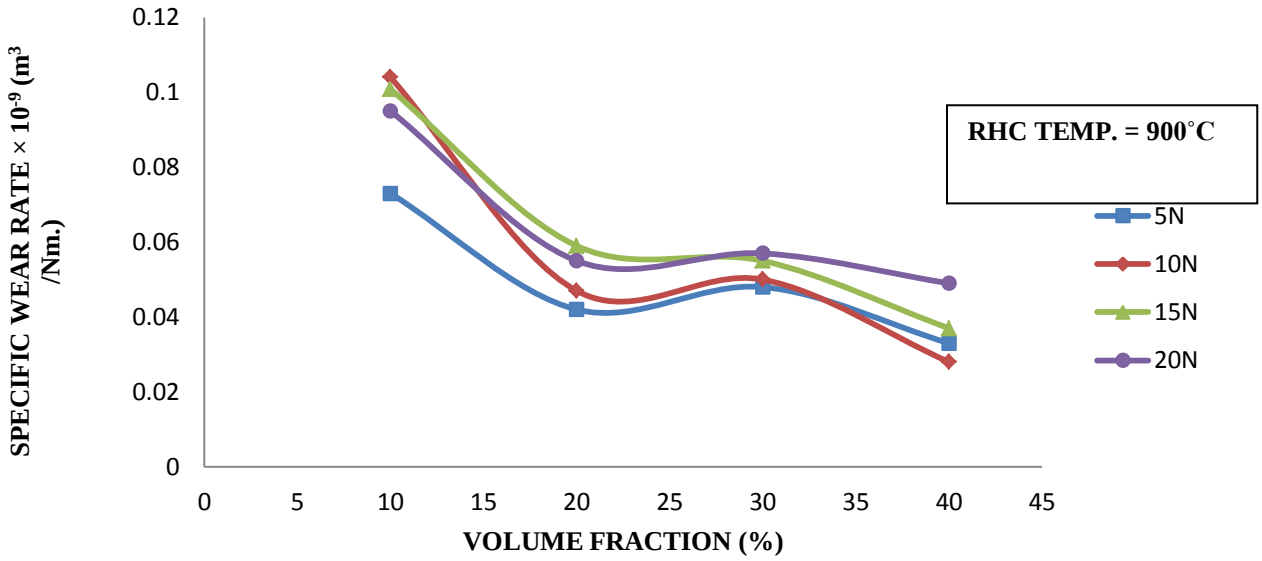

Figure-3.21 Variation of specific wear rate with different volume fraction of reinforcement at different normal load, carbonization temperature 900˚c.

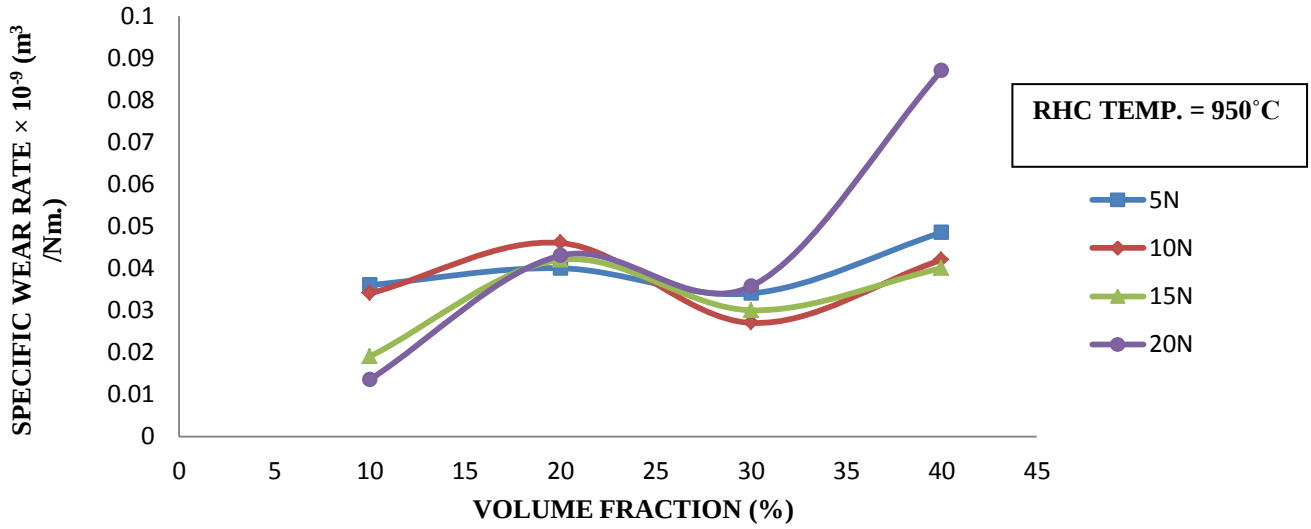

Figure-3.22 Variation of specific wear rate with different volume fraction of reinforcement at different normal load, carbonization temperature 950˚c

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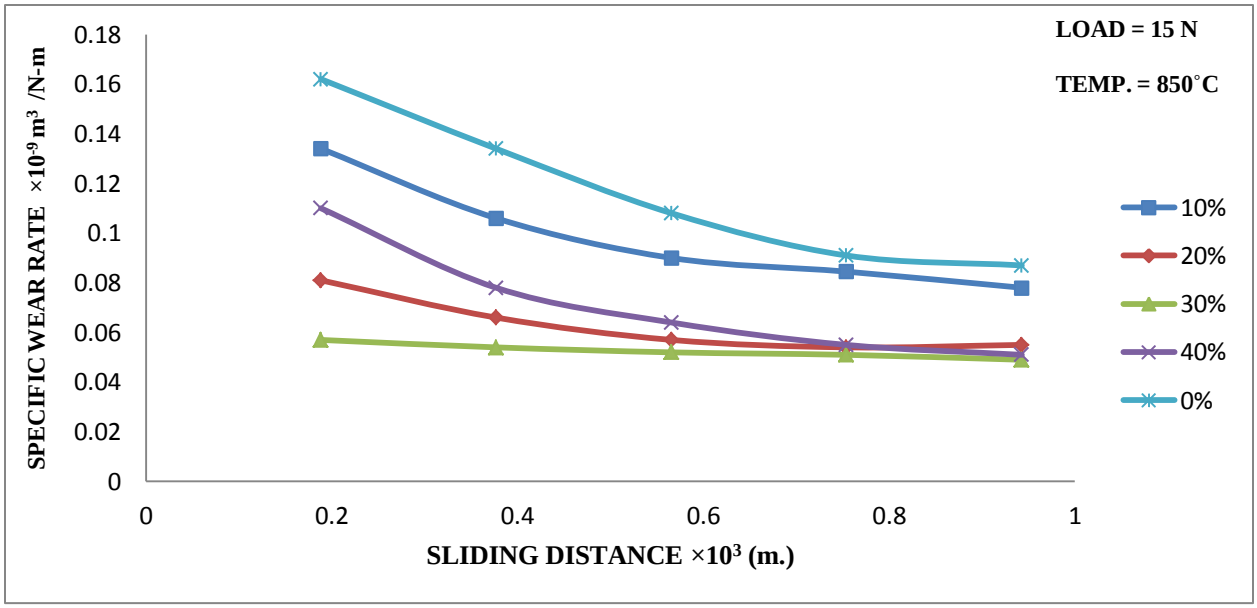

Figure-3.23 Variation of specific wear rate with sliding distance at sliding velocity of v=0.633 m/s and at different volume fraction at 15N normal load.

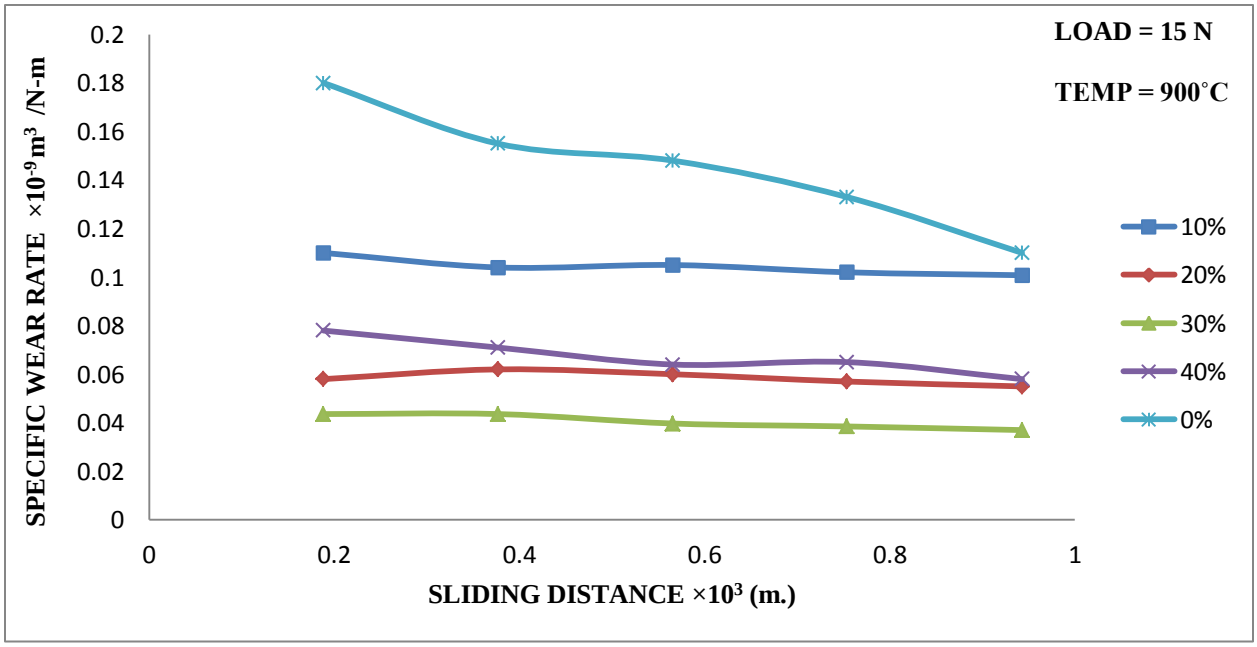

Figure-3.24 Variation of specific wear rate with sliding distance at sliding velocity of v=0.633 m/s and at different volume fraction at 15N normal load.

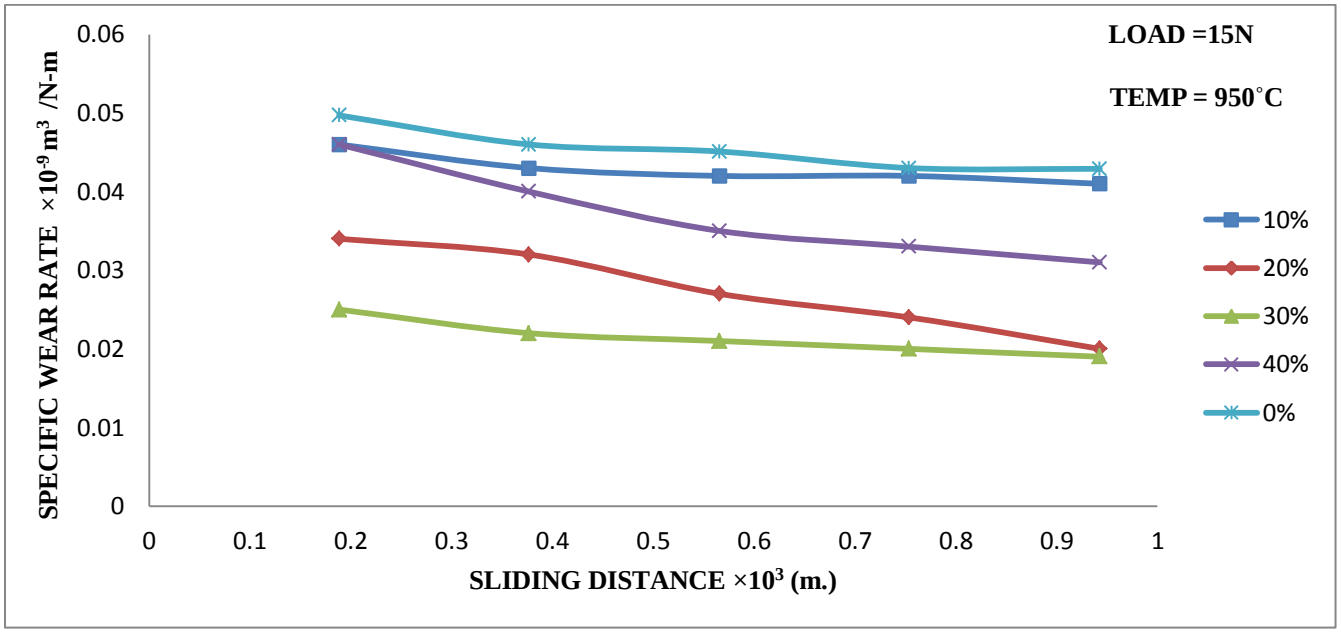

Figure-3.25 Variation of specific wear rate with sliding distance at sliding velocity of v=0.633 m/s and at different volume fraction at 15N normal load.

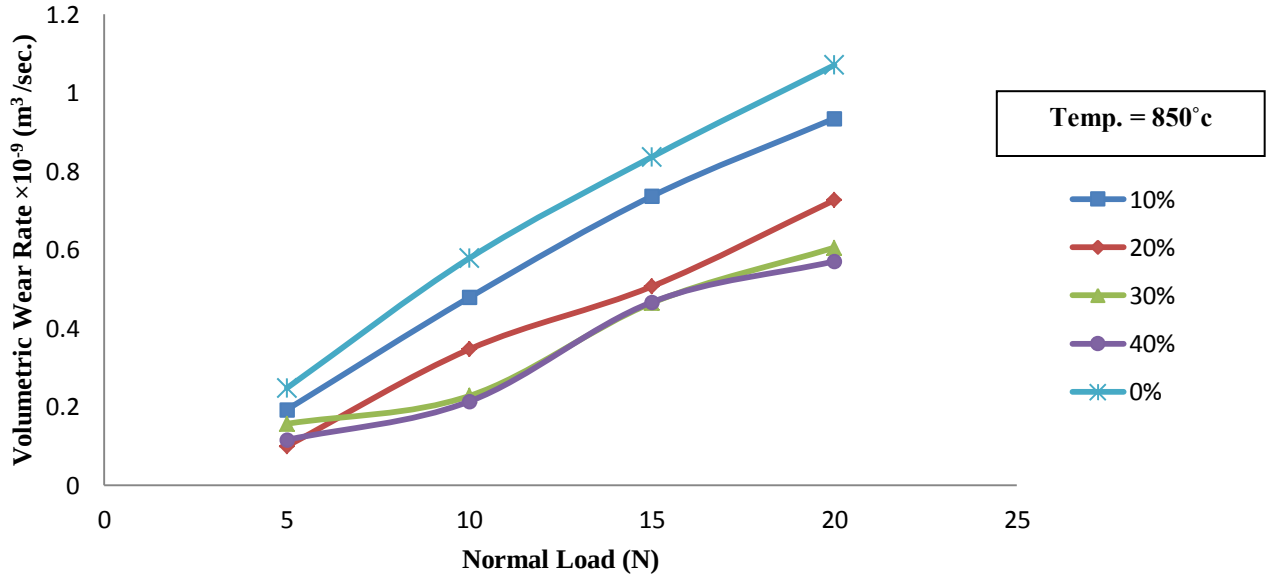

Figure-3.26 Variation of volumetric wear rate with normal load at sliding velocity of v=0.633 m/s and at different volume fraction.

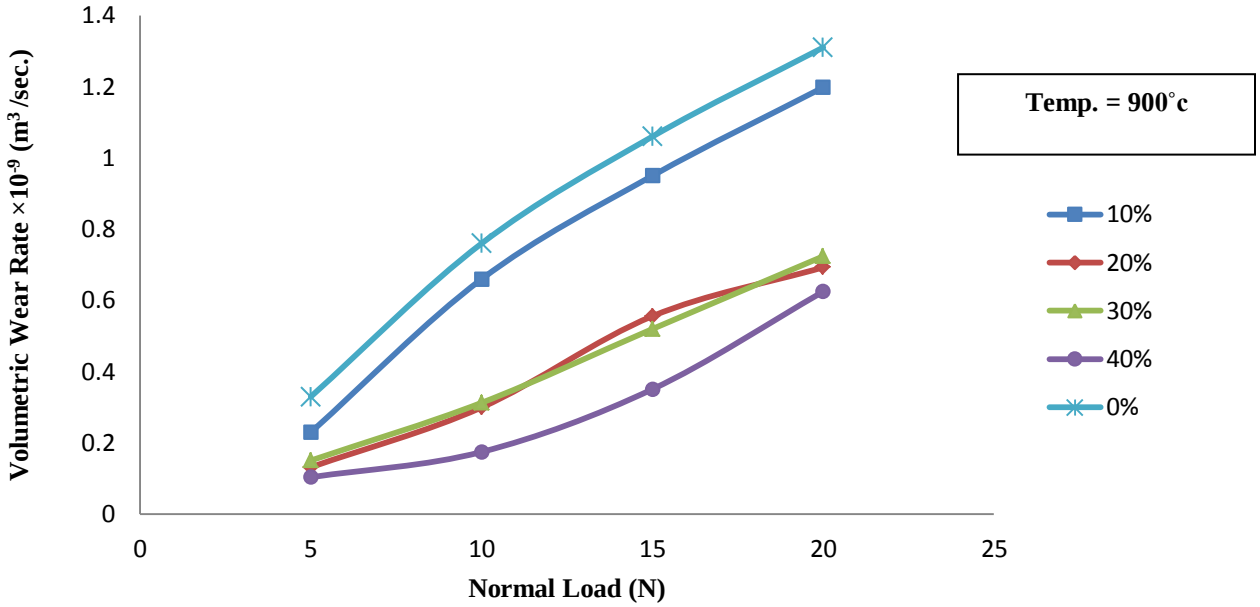

Figure-3.27 Variation of volumetric wear rate with normal load at sliding velocity of v=0.633 m/s and at different volume fraction.

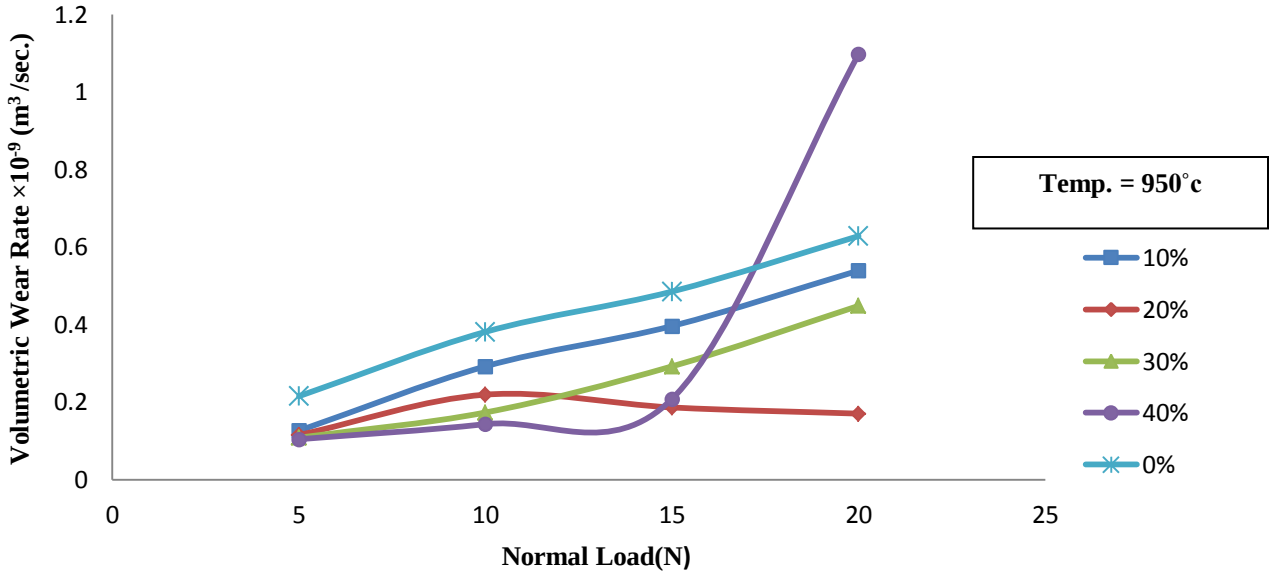

Figure-3.28 Variation of volumetric wear rate with normal load at sliding velocity of v=0.633 m/s and at different volume fraction.

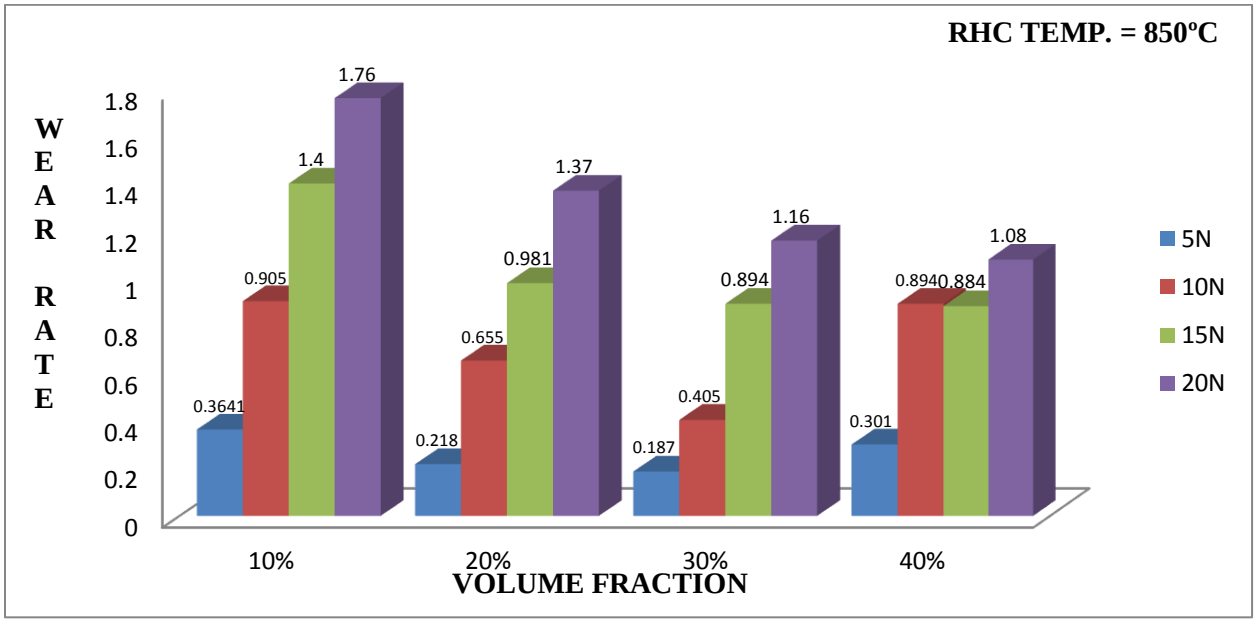

Figure-3.29 Variation of wear rate with volume fraction at different normal load with carbonization tempreture 850˚c.

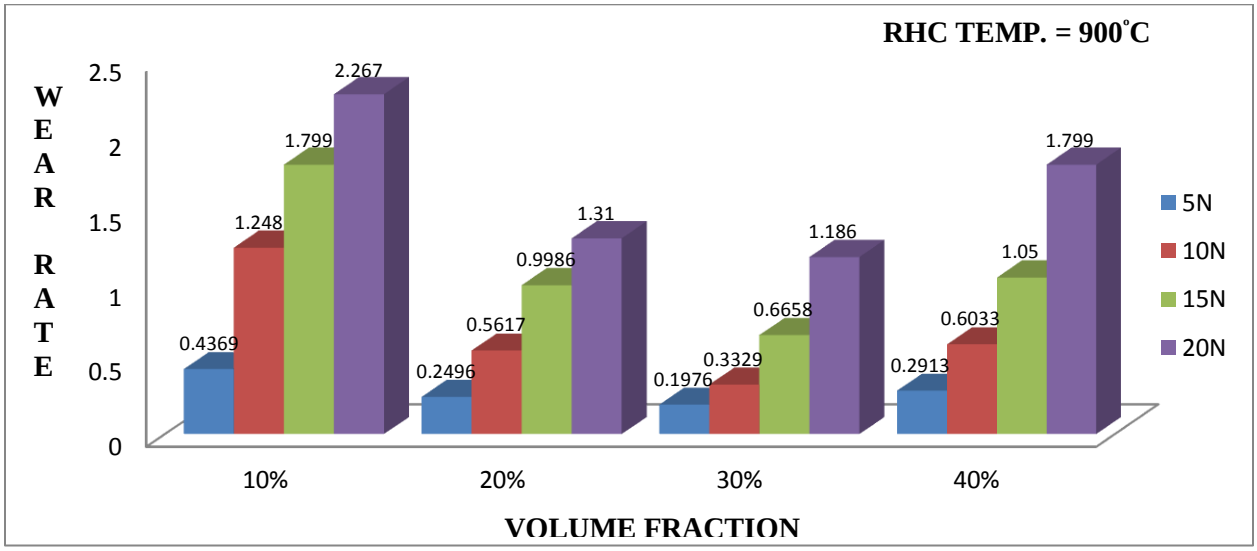

Figure-3.30 Variation of wear rate with volume fraction at different normal load with carbonization tempreture 900˚c.

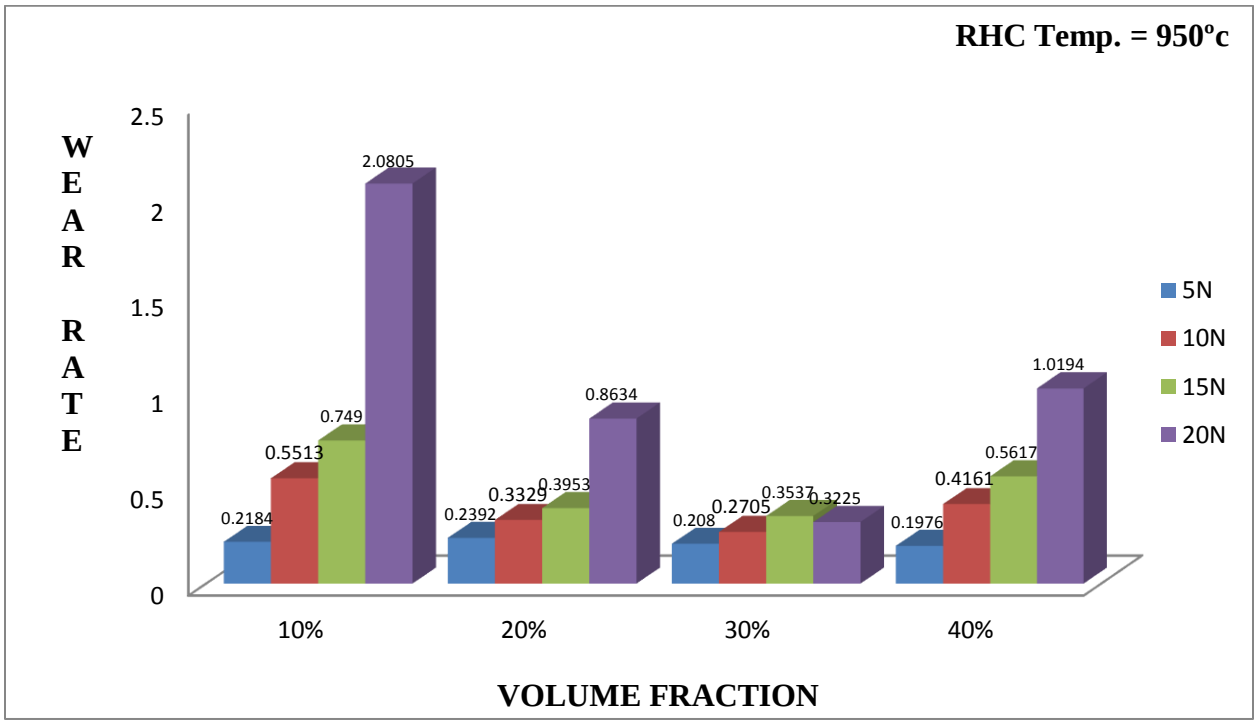

Figure-3.31 Variation of wear rate with volume fraction at different normal load with carbonization tempreture 950˚c.

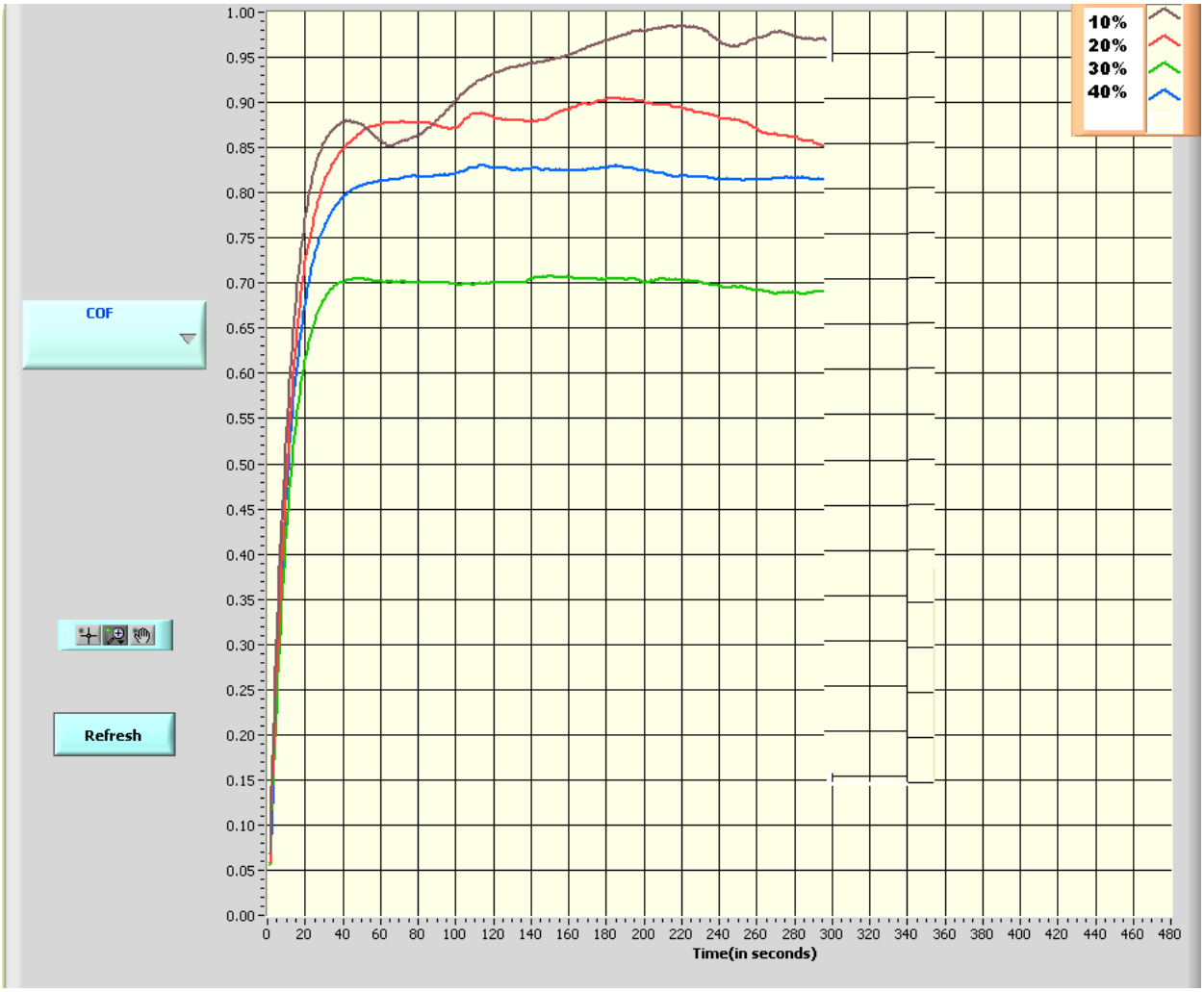

Figure-3.32 Plots between the friction coefficients and time for different composites (10vol% to 40vol% fiber reinforced epoxy composite) at 20N applied normal load, 0.62832m/s sliding velocity and fiber made at 850°c .

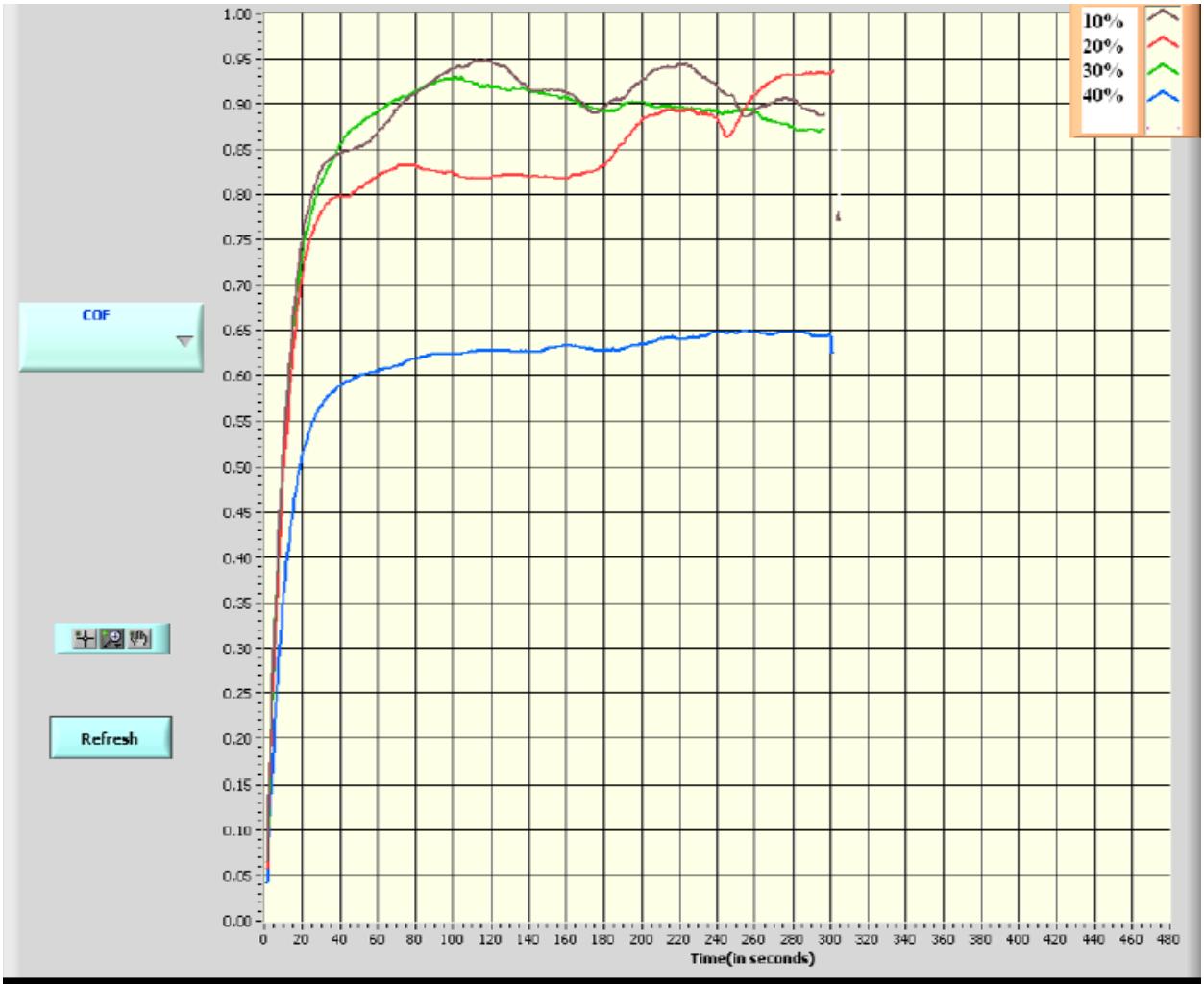

Figure-3.33 Plots between the friction coefficients and time for different composites (10vol% to 40vol% fiber reinforced epoxy composite) at 20N applied normal load, 0.62832m/s sliding velocity and fiber made at 900°c .

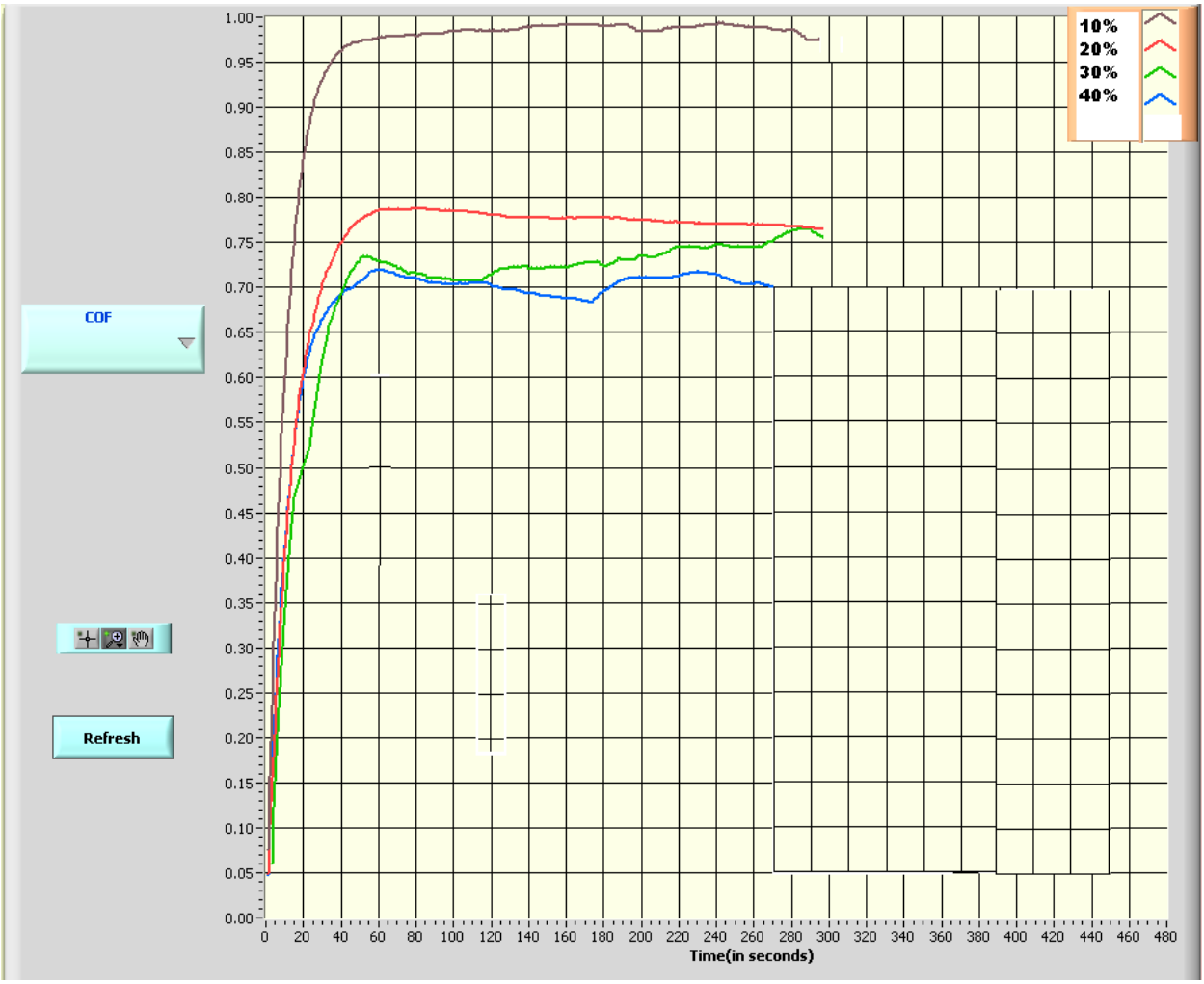

Figure-3.34 Plots between the friction coefficients and time for different composites (10vol% to 40vol% fiber reinforced epoxy composite) at 20N applied normal load, 0.62832m/s sliding velocity and fiber made at 950°c .

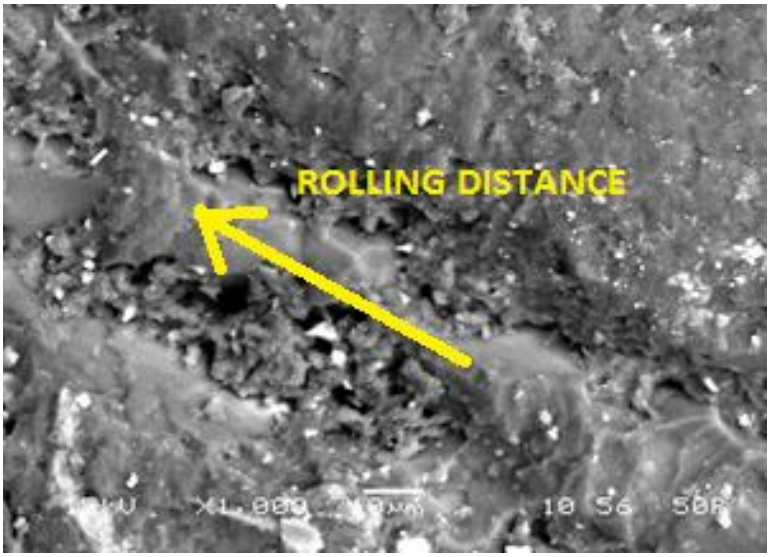

Figure -3.35 (a)

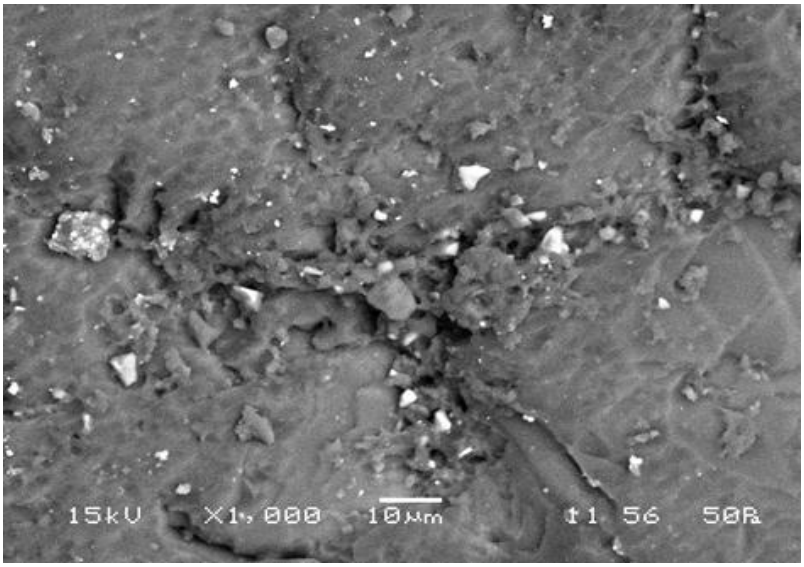

Figure -3.35 (b)

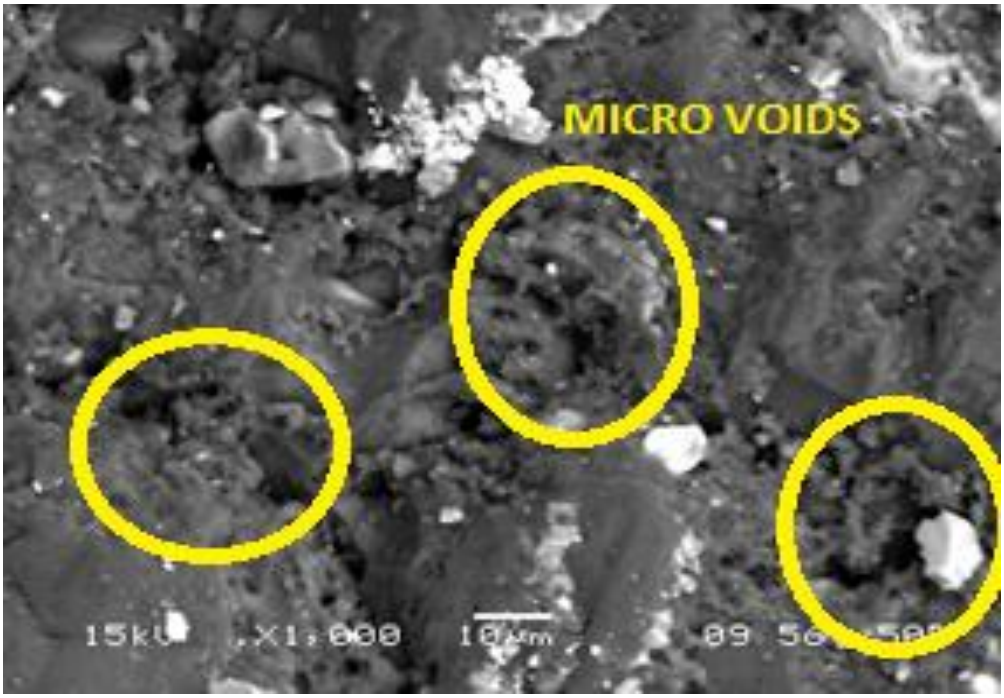

Figure-3.35 (c)

Figure-3.35 Scanning electron micrograph of worn surface of tested composite samples; (a) 30% reinforcement made at 850˚c, (b) 30% reinforcement made at 900˚c, (c) 30% reinforcement made at 950˚c.

Chapter 4

CONCLUSION

CHAPTER-4

4.1 CONCLUSIONS

The following conclusions have been drawn from the above studies

1. The Rice husk one of the byproduct of rice mill can successfully be utilized to produce composite by suitably bonding with resin for value added product.
2. Incorporation of RH Char in to epoxy, can significantly reduce abrasive wear loss. The optimum wear resistance property was obtained at the fiber content of 30 weight fraction.
3. The specific wear rate of composite decreases with the increases of sliding distance because the space between the abrasive filled by the debris, which reduced the depth of penetration abrasive particle in to the composite sample.
4. The co-efficient of friction decreases as the volume fraction of reinforcement (RH ceramic) increases.
5. Minimum wear rate is observed for 30% volume of reinforcement at 950˚c carburized temperature with 15N load.

4.2 RECOMMENDATION FOR FURTHER RESEARCH

- It is known that fiber surface modification increases the bonding strength between fiber and the matrix. Therefore fiber modification can be done to improve the strength.
- In addition to tribological testing mechanical tests should be carried out.
- These composites are showing promising results for wear applications. Therefore this work can be further extended to other tribological tests.

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Chapter 5

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REFERENCES

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